

FOOD PROCESSING REVIEW No. 10

# *Animal Feeds*

*M. Gutcho*

CFTRI-MYSORE



3588

Animal feeds 197..

**ndc**



## CHEMICAL PROCESS REVIEWS

Chemical Process Reviews are a series of comprehensive up-to-date technological studies of specific chemical products. Devoted primarily to production techniques, they are down-to-earth practical publications for managerial, research, development, engineering, and marketing executives in the chemical industry.

|   |      |
|---|------|
| Synthetic Fibers from Petroleum; Sittig - \$35                                    | 1967 |
| Polyolefin Processes; Sittig - \$35   | 1967 |
| Stereo-Rubber and Other Elastomer Processes; Sittig - \$35                        | 1967 |
| Industrial Gases - Manufacture and Applications; Sittig - \$35                    | 1967 |
| Pesticide Production Processes; Sittig - \$35                                     | 1967 |
| Plastic Films from Petroleum Raw Materials; Sittig - \$35                         | 1967 |
| Catalysts and Catalytic Processes; Sittig - \$35                                  | 1967 |
| Combine Hydrocarbons and Nitrogen for Profit; Sittig - \$35                       | 1967 |
| Phosphoric Acid by the Wet Process; Noyes - \$35                                  | 1967 |
| Combine Hydrocarbons and Halogens for Profit; Sittig - \$35                       | 1968 |
| Combine Hydrocarbons and Oxygen for Profit; Sittig - \$35                         | 1968 |
| Olefins - Manufacture and Derivatives; Sittig - \$35                              | 1968 |
| Urea Process Technology; Powell - \$35  | 1968 |
| Electro-Organic Chemical Processing; Mantell - \$35                               | 1968 |
| Diolefins - Manufacture and Derivatives; Sittig - \$35                            | 1968 |
| Controlled Release Fertilizers; Powell - \$35                                     | 1968 |
| Titanium Dioxide and Titanium Tetrachloride; Powell - \$35                        | 1968 |
| Aromatics - Manufacture and Derivatives; Sittig - \$35                            | 1968 |
| Paraffins and Cycloparaffins - Manufacture and Derivatives; Sittig - \$35         | 1968 |
| Hydrogen Peroxide Manufacture; Powell - \$35                                      | 1968 |
| Acetylene Processes and Products; Sittig - \$35                                   | 1968 |
| Carbon Black Technology; Powell - \$35  | 1968 |
| Alcohols, Polyols, and Phenols - Manufacture and Derivatives; Sittig - \$35       | 1968 |
| Monosodium Glutamate and Glutamic Acid; Powell - \$35                             | 1968 |
| Polyacetal Resins, Aldehydes, and Ketones - Processes and Products; Sittig - \$35 | 1968 |
| Hydrazine Manufacturing Processes; Powell - \$35                                  | 1968 |
| Air Pollution Control; Sittig - \$35  | 1968 |
| Nitric Acid Technology; Powell - \$35   | 1968 |
| Practical Detergent Manufacture; Sittig - \$35                                    | 1968 |
| Water Pollution Control; Sittig - \$35  | 1969 |
| Synthetic Leather from Petroleum; Sittig - \$35                                   | 1969 |
| Amines, Nitriles, and Isocyanates - Processes and Products; Sittig - \$35         | 1969 |
| Chlorine and Caustic Soda Manufacture; Powell - \$35                              | 1969 |
| Alkali Metal Phosphates; Ranney - \$35  | 1969 |
| Ammonium Phosphates; Ranney - \$35  | 1969 |
| Photochemical Processes; Albertson - \$35   | 1969 |
| Citric Acid Production Processes; Noyes - \$24                                    | 1969 |
| Fibrinolytic Enzyme Manufacture; Rubel - \$24                                     | 1969 |
| Vitamin E Manufacture; Rubel - \$24   | 1969 |
| Vitamin B <sub>12</sub> Manufacture; Noyes - \$35                                 | 1969 |
| Radiation Chemical Processing; Whiting - \$35                                     | 1969 |



# **Animal Feeds**

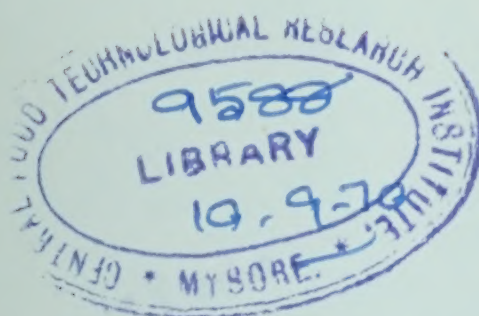
## **1970**

**M. Gutcho**

**Thirty-Five Dollars**

**NOYES DATA CORPORATION**  
Park Ridge, New Jersey, U.S.A.

9588



J2

~~LO~~

N50

Copyright © 1970 by Noyes Data Corporation

No part of this book may be reproduced in any form,  
without permission in writing from the Publisher.

Library of Congress Catalog Card Number: 77-104725

SBN: 8155-0297-4

Printed in the United States

CFTRI-MYSORE



9588

Animal feeds 197.



## FOREWORD

The detailed, descriptive process information in this Food Processing Review is based on U.S. patents since 1960, relating to the production of animal feeds.

This book serves a double purpose in that it supplies detailed technical information, and can be used as a guide to the U.S. patent literature in this field. By indicating only information that is significant, and eliminating much of the legal jargon in the patents, this book then becomes an advanced industrially oriented review of processes to produce animal feeds.

The U.S. patent literature is the largest and most comprehensive collection of technical information in the world. There is more practical, commercial, timely process information assembled here than is available from any other source. The technical information obtained from the patent literature is extremely reliable and comprehensive; sufficient information must be included to avoid rejection for "insufficient disclosure".

The patent literature covers a substantial amount of information not available in the journal literature. The patent literature is a prime source of basic commercially utilizable information. This information is overlooked by those who rely primarily on the periodical journal literature. It is realized that there is a lag between a patent application on a new process development and the granting of a patent, but it is felt that this may roughly parallel or even anticipate the lag in putting that development into commercial practice.

These Reviews are bound in paper in order to close the time gap between "manuscript" and "completed book". Industrial technology is progressing so rapidly that hard cover books do not always reflect the latest developments in a particular field, due to the longer time required to produce a hard cover book.

The Table of Contents is organized in such a way as to serve as a subject index. Other indexes by company, inventor, and patent number help in providing easily obtainable information.



## FOREWORD

The author, Elizabeth, is a member of the American Psychological Association and is a member of the American Psychological Association.

The book is a study of the life of a woman who was a member of the American Psychological Association and is a member of the American Psychological Association.

The author, Elizabeth, is a member of the American Psychological Association and is a member of the American Psychological Association.

The author, Elizabeth, is a member of the American Psychological Association and is a member of the American Psychological Association.

The author, Elizabeth, is a member of the American Psychological Association and is a member of the American Psychological Association.

The author, Elizabeth, is a member of the American Psychological Association and is a member of the American Psychological Association.



# CONTENTS

|   |    |
|---|----|
| 1. INTRODUCTION   | 1  |
| 2. <u>FORAGE AND FODDER</u>                                   | 2  |
| Preparation and Preservation of Silage                        | 2  |
| Preparation of Silage Using Malt                              | 2  |
| Use of Malt Diastase and Sodium Sulfate                       | 3  |
| Use of Aqueous Ammoniated Superphosphate                      | 4  |
| Antioxidant Plus Enzyme Combination                           | 5  |
| Bacterial Fermentation with <i>Pediococcus Cerevisiae</i>     | 7  |
| Streptomycin Plus Streptomycin Resistant Lactic Acid Bacteria | 7  |
| Zinc Bacitracin Plus Molasses                                 | 9  |
| Sodium Tripolyphosphate and Sodium Hexametaphosphate          | 10 |
| Addition of Dry Substances Containing Starch and Protein      | 10 |
| Fermented Feed Substance Without Green Fodder                 | 11 |
| Storage of Grain Under Carbon Dioxide                         | 12 |
| Drying of Forage Material                                     | 13 |
| Fast Forage Drying Technique                                  | 13 |
| Increasing Protein Content of Forage Material                 | 15 |
| Reducing Carotene Loss During Storage                         | 16 |
| Preserving Hay  | 17 |
| Meal from Forage Plants                                       | 18 |
| Other Fodders   | 19 |
| Fodder from Chopped Straw                                     | 19 |
| Silica Substitute for Roughage in Feed                        | 19 |
| Hydroponically Produced Vegetal Fodder                        | 21 |
| Forage Pellets  | 24 |
| Enzyme Treated Starch Binder for Forage Pellets               | 24 |
| Wafer from Forage Material                                    | 26 |
| Feed Pellet of Unchopped Hay                                  | 27 |
| Pelleting Hay at Low Pressures                                | 29 |
| Pelleting Roughage Crops                                      | 30 |
| Pelleted Feed Containing Fresh Green Forage                   | 33 |
| Feed Licks and Blocks   | 34 |
| Chalk-Glucose Licks   | 34 |
| Protein Feed Block  | 35 |
| Nutrient Feed Blocks  | 36 |
| Detoxifying Plant Material                                    | 38 |
| Removing Toxins from <i>Agave Lecheguilla</i>                 | 38 |
| Treating Oleaginous Substances to Reduce Growth Inhibitors    | 39 |
| Detoxified Meal from <i>Crambe Abyssinica</i> Seed            | 40 |
| Microbiological Decontamination of Aflatoxin                  | 40 |
| 3. <u>FATS AND OILS</u>                                       | 43 |
| Refining of Vegetable Glyceride Oils                          | 43 |



## Contents

|   |        |
|---|--------|
| Use of Sodium Carbonate   | 43     |
| Removal of CO <sub>2</sub> Before Oil-Soapstock Separation          | 44     |
| Oil Extraction with Aqueous Ammonia                                 | 47     |
| Using Sequentially Caustic and Soda Ash                             | 48     |
| Low-Excess Soda Ash Soapstock                                       | 48     |
| Soapstocks in Feed Products   | 49     |
| Detoxified Cottonseed Oil Soapstock                                 | 50     |
| Soapstock Meal Blend  | 50     |
| Lecithin Products from Soapstocks                                   | 51     |
| Dry Additives Prepared from Soapstock                               | 52     |
| Soaps in Animal Feeds   | 52     |
| High Fat Oilseed Meals  | 53     |
| Dry Oilseed Meal  | 53     |
| Thermal Pressure Preparation of High Fat Meal                       | 55     |
| Oilseed Meal Dry Pellet   | 55     |
| Products from Condensate of Vegetable Oil Deodorization             | 56     |
| Tocopherol Rich Stable Deodorizer Distillate                        | 56     |
| Vegetable Oil, Animal Fat, Crude Well Oil Composition               | 57     |
| Powdered Absorbent Feed Materials Containing Crude Hot Well Oil     | 59     |
| Vegetable Oil Emulsions   | 60     |
| Vitamin Enriched Vegetable Oil Emulsion                             | 60     |
| Vegetable Oil-Lecithin-Tocopherol Composition                       | 60     |
| Fats in Feeds   | 62     |
| Digestible, Discrete, Fat Flakes                                    | 63     |
| Choline-Fat Composition   | 63     |
| High Fat Content Food Pellets                                       | 64     |
| <br>4. <u>MOLASSES AND FLAVORINGS</u>                               | <br>65 |
| <u>Molasses</u>   | 65     |
| Non-Hygroscopic Molasses Product                                    | 65     |
| Dried Molasses Product  | 66     |
| Molasses on an Oat Hulls Carrier                                    | 66     |
| Molasses-Vermiculite Product  | 68     |
| Fibrous Absorbent Flake from Hulls                                  | 68     |
| Pelleted Molasses-Containing Feed                                   | 71     |
| Liquid Ruminant Supplement  | 71     |
| Stable Feed Emulsions Containing Molasses                           | 73     |
| Prevention of Gel Formation in Molasses-Phosphoric Acid Supplements | 74     |
| Use of Non-Phosphatic Acid  | 74     |
| Addition of Alkali Metal Polyphosphate                              | 76     |
| Ammoniated Sugars   | 77     |
| Ammoniated Low Grade Sugar Products                                 | 77     |
| Ammoniated Sweetened Feed   | 79     |
| <u>Flavorings</u>   | 81     |
| Antioxidant Flavoring   | 81     |
| Flavored Non-Carbohydrate Sweetener                                 | 82     |
| Concentrated Flavoring Premix                                       | 84     |
| <br>5. <u>ESTROGENS AS GROWTH STIMULATORS</u>                       | <br>86 |
| Compounds from Cultures of <i>Gibberella</i>                        | 86     |
| Gibberellin   | 86     |
| Estrogenic Substance from <i>Gibberella Zeae</i> Culture            | 87     |
| Estrogenic Compounds from Fermentation Estrogenic Substance         | 89     |
| Coumarone Compounds   | 94     |
| Coumestrol Ethers   | 94     |
| Other Coumarone Derivatives   | 95     |
| Diethylstilbestrol (DES) Premix                                     | 95     |
| Compact Diethylstilbestrol Premix                                   | 95     |
| Improving Stability of Diethylstilbestrol-Containing Premix         | 97     |
| Safe Handling of Estrogenic Feed Supplements                        | 98     |



## Contents

|  |         |
|--|---------|
| Synergistic Effect of DES  | 99      |
| Estrogen Plus 1-Methyl-2-Mercapto-3-Carbethoxy Imidazole               | 99      |
| Estrogen Plus 2-Mercaptoimidazole                                      | 100     |
| Estrogen Plus Steroidal Sapogenin                                      | 101     |
| Other Hormones   | 101     |
| 9 $\alpha$ -Fluoro-16 $\alpha$ -Methylprednisolone                     | 101     |
| Glucocorticoids  | 103     |
| Mass Hormonization   | 103     |
| <br>6. <u>ANTIBIOTICS AS ANABOLIC STIMULATORS</u>                      | <br>106 |
| <u>Stabilizing Bacitracin Feedstuffs</u>                               | 106     |
| Use of Manganese   | 106     |
| Manganese Plus Reducing Agent  | 107     |
| Use of Cobalt  | 107     |
| Ligin-Bacitracin Complex   | 108     |
| Bacitracin Adsorbate (Cation Exchange Resin)                           | 109     |
| Precipitation of Bacitracin Salts on Inert Support                     | 110     |
| Other Bacillus Strains   | 110     |
| Growth Factors from Specific Strains of <i>Bacillus Subtilis</i>       | 110     |
| Growth Promotor from <i>Bacillus Licheniformis</i>                     | 113     |
| Growth Factors from <i>Bacillus Subtilis</i> and <i>Bacillus Natto</i> | 114     |
| Insect Resistant Animal Feeds  | 115     |
| Stabilizing Tetracycline Feedstuffs                                    | 116     |
| 7-Chloro-4-Epitetracycline   | 116     |
| Maintaining Feed Supplement pH at 8.5 to 11.5                          | 117     |
| pH Adjustments to Improve Stability                                    | 118     |
| Addition of Aluminum Salts Prior to Harvesting Mash                    | 119     |
| Heat Treatment Prior to Harvesting Mash                                | 120     |
| Tetracycline in Feed Supplements                                       | 121     |
| Forms of Calcium and Phosphorus in Tetracycline Diets                  | 121     |
| Antibiotic, Estrogen, Anthelmintic Combination                         | 122     |
| Antibiotic Plus Stilbene Derivative                                    | 124     |
| Tetracycline, Sulfonamide, Penicillin Combination                      | 126     |
| Antibiotics from Other Streptomyces                                    | 127     |
| Viridogrisein Plus Griseoviridin                                       | 127     |
| Staphylomycin  | 128     |
| Spiramycins  | 129     |
| Neomycin Resin Adsorbate   | 130     |
| Moenomycin   | 131     |
| Phytoactin and Phytostreptin   | 131     |
| Oleandomycin   | 133     |
| Oleandomycin Resin Adsorbate   | 134     |
| Spectinomycin  | 135     |
| Combination of Spectinomycin and Lincomycin                            | 136     |
| Antimicrobials   | 137     |
| Alkyl-2-Haloacetoacetates  | 137     |
| Acroleins and Beta-Methoxypropionaldehyde                              | 138     |
| Miscellaneous  | 139     |
| Curvularin   | 139     |
| Hydrolyzates of Mycelia of Actinomycetes <i>Nocardia Rugosa</i>        | 140     |
| <br>7. <u>ANTIOXIDANTS IN FEEDS</u>                                    | <br>142 |
| <u>"Santoquin" Absorbed on Mineral-Vegetable Powder</u>                | 142     |
| Activated Dihydroquinolines  | 143     |
| Dihydroquinolines Activated with 8-Hydroxyquinoline                    | 144     |
| Dihydroquinolines Activated with Sulfur-Containing Amino Acids         | 144     |
| Dihydroquinolines Activated with Substituted Anilines                  | 145     |
| Dihydroquinolines Activated with Nitrogen-Containing Compounds         | 145     |
| Dihydroquinoline Acid Salts  | 146     |



|   |     |
|---|-----|
| 12. FEED FOR SWINE  | 268 |
| Prevention of Iron Deficiency Anemia in Baby Pigs           | 268 |
| Sweetened Iron Supplement                                   | 270 |
| Humus-Soft Phosphate-Colloidal Clay-Iron Supplement         | 270 |
| Iron-Sphagnum Moss Preparation                              | 271 |
| Feeding Sow Iron Agent                                      | 273 |
| Promoting Leanness of Carcass in Swine                      | 273 |
| Nitroalcohols and Nitrocarbarnates                          | 274 |
| Ditnitro Compounds  | 274 |
| Nicotine  | 275 |
| Hypoglycemic Sulfonylurea                                   | 277 |
| Other   | 277 |
| Dry Feed for Weaning Pigs                                   | 277 |
| Mercaptoimidazole Compounds as Growth Promoters             | 278 |
| 13. PET AND OTHER FEEDS                                     | 280 |
| Pet Food  | 280 |
| Hydratable Food Forming Own Gravy                           | 280 |
| Semi-Moist Marbelized Meaty Food                            | 281 |
| High Acid Content Palatable Dog Food                        | 283 |
| Pelleted Dog Food Using Raw Cereal Grains                   | 284 |
| Intermediate Moisture Type Food                             | 287 |
| Fat Coated Feed Annuli                                      | 288 |
| Acetamide Flavor Enhancer                                   | 290 |
| Compositions for Birds                                      | 291 |
| Diet Supplement for Caged Pet Birds                         | 291 |
| Introducing Physiologically Active Material into Bird Seed  | 292 |
| Molded Cuttlebone   | 292 |
| Pecking Stone for Birds                                     | 293 |
| Fish Baits  | 294 |
| Water-Soluble Fish Bait                                     | 294 |
| Dough Balls   | 298 |
| Freeze Dried Unified Mass                                   | 298 |
| Silkworm Feeds  | 300 |
| Eating-Inducing Compositions                                | 300 |
| Feed Intake Promoting Factor                                | 300 |
| Propionic Acid as Growth Accelerator                        | 301 |
| Sterile Animal Food   | 301 |
| Sterilizing Food in Sealed Container                        | 301 |
| Sterile Pelleted Food                                       | 303 |
| 14. UTILIZATION OF INDUSTRIAL WASTE AND <u>BY-PRODUCTS</u>  | 307 |
| Feed Supplements from Poultry Waste                         | 307 |
| Offal Processing at 60°F.                                   | 307 |
| Foodstuff from Poultry Offal                                | 308 |
| Food for Carnivorous Animals from Poultry Viscera           | 310 |
| Supplement from Combination of Offal and Feathers           | 312 |
| Low Ash-Content Supplement from Poultry Bones               | 314 |
| Products from Meat Industry                                 | 316 |
| Blood Meal from Hydrolysis of Whole Blood                   | 316 |
| Blood Meal from Acid Hydrolysis of Whole Blood              | 317 |
| Economical Blood Meal Process                               | 318 |
| Feed Product from Animal Manure                             | 319 |
| Protein, Fat and Bone Meal from Dry Rendered Tankage        | 321 |
| Feed Supplement from Tannery Fleshings                      | 323 |
| Dry Free Flowing Food Supplement from Animal By-Products    | 326 |
| High Fat Content Feed Product                               | 327 |
| Feed Supplements from Fish Industry                         | 328 |
| Nonhygroscopic Fish Solubles Product on Vermiculite Carrier | 328 |



## Contents

|  |     |
|--|-----|
| Pellets from Fish Meal and Condensed Fish Solubles     | 329 |
| Fish Cake from Rough Fish                              | 330 |
| Stable Fish Meal                                       | 332 |
| Pet Food from Clam Waste                               | 333 |
| Miscellaneous  | 333 |
| Stabilized Fermentation Broths                         | 333 |
| Salt Blocks from Potash Refining Wastes                | 336 |
| Spent Sulfite Wood Liquor as Binder for Pelleted Feeds | 339 |
| Starch Hydrolyzate Liquor-Vegetable Meal Dry Feedstuff | 340 |
| Use of Bleaching Earth from Decolorizing of Fatty Oils | 340 |
| Livestock Feed from Citrus Waste                       | 341 |
| COMPANY INDEX  | 344 |
| INVENTOR INDEX   | 346 |
| U.S. PATENT NUMBER INDEX                               | 350 |







## INTRODUCTION

Providing food for the world has become more vital than ever. In the United States meat is important both as a food product, and to the national economy. The trend in modern livestock production is toward producing animals with a rapid rate of growth. This permits early marketing of such animals, which results in great savings to the animal producer. The faster a chicken reaches the egg laying stage, the faster a profit is realized. Not too many years ago it took 11 weeks from hatching to develop a 3 lb. broiler chicken using 3 1/4 lb. of feed per pound of weight gain. Now 9 weeks after hatching a broiler of that size is obtained with a consumption of only 2 1/2 lb. of food per pound of weight gain.

Extensive study of the nutritional requirements of animals has led to the development of nutritionally balanced feeds which give the proper proportion of protein, carbohydrates, minerals and vitamins. There is continuous research and investigation toward the development of new and accessory factors for feed compositions and supplements with a view toward achieving still further gains from the standpoint of rate of growth and amount of growth per unit of food. These accessory factors are not necessary from the nutritional standpoint, but act as growth stimulators.

Many of the processes in this book are concerned with just such growth promoting additives. Some deal with methods for the preparation of food compositions from conventional or new sources. In others, primary interest is in preparing supplements which are more digestible, more acceptable to animals. Processes for improving the stability of feeds to time, temperature and moisture are also covered.

## FORAGE AND FODDER

### PREPARATION AND PRESERVATION OF SILAGE

One of the most important problems with which the farmer is continually faced is the storage and preservation of green forage. The production of silage from plant roughage such as grasses, legumes and other forage crops including bluegrass, orchardgrass, alfalfa, clover, corn, sorghum, oats and fescue is an ever increasingly popular method of harvesting, storing and preserving such valuable feedstuffs. This is because there are distinct advantages in making silage from forage crops over curing them as hay, the relative independence of the silage making process on weather, the superior feeding value of silage over ordinary hay, the greater palatability of silage, the ease of feeding silage with little or no waste and the decreased storage space necessary for the more compact silage.

The silage making process comprises essentially chopping and packing green forage into a storage container, usually a cylindrical silo or pit in the ground, and allowing fermentation to take place with production of lactic acid. When the chopped green forage is tightly packed in the silo, the microorganisms utilize the nutrients from expressed juices and ferment them into acidic substances. The types of microorganisms present in the packed material, the supply of fermentable nutrients, the level of entrapped air in the forage, and the type and rate of fermentation determine to a large extent the quality of the silage produced. For good silage it is important that the fermentation produce mainly lactic acid and that this fermentation take place rapidly. These processes relate to new developments in the preparation and preservation of silage.

### Preparation of Silage Using Malt

C.M. Hollenbeck and J.M. Sheneman (U.S. Patent 3,033,685; May 8, 1962; assigned to Wisconsin Malting Co.) provide a process for making silage from plant roughage and forage crops such as grasses, legumes and/or corn which insures rapid and proper production of lactic acid within the first few days by incorporating a small amount of barley malt or malt by-products into the chopped forage material during packing into the container, i.e., silo.

Although the malt as such can be added to the forage material directly, it is sometimes desirable to first prepare a premix of the malt or malt by-product with a diluent such as chopped forage itself, either green or dried, or ground corn cobs, cracked or ground grains, dried sugar beet pulp, soybean meal, dried molasses or the like. The malt or malt by-product content



of such premixes can be any amount desired but would generally be from 10 to 75% by weight.

Example: Samples of a mixture of grasses and legumes were ensiled by the laboratory procedure, with and without 2% ground malt by weight of the green material. The index of silage quality in these experiments is the rate of drop in pH, i.e., the faster the drop in pH the better the silage quality. It is readily noted that the forage containing the ground malt rapidly and continually developed acid materials. The pH dropped to below 4.5 in 5 days with very little fluctuation, up and down, on continued fermentation. The odor of the malt treated samples was pleasant, while the odor of the controls became putrid and spoiled.

| <u>Days of Fermentation</u> | <u>pH</u>      |                |
|-----------------------------|----------------|----------------|
|                             | <u>Control</u> | <u>2% Malt</u> |
| 0                           | 6.20           | 6.20           |
| 5                           | 5.05           | 4.45           |
| 7                           | 4.80           | 4.35           |
| 13                          | 4.90           | 4.45           |
| 26                          | 5.20           | 4.30           |
| 28                          | 4.90           | 4.25           |
| 36                          | 5.15           | 4.30           |

## Use of Malt Diastase and Sodium Sulfate

J.G. Forest, and E.J. Czarnetzky (U.S. Patent 3,184,314; May 18, 1965; assigned to International Stock Food Corporation) have developed a method for preserving silage and more particularly to a method for inhibiting the loss of nutrients in silage using a mixture of malt diastase and sodium sulfate.

Example 1: Tests were conducted with peavine silage with varying amounts of a mixture of sodium sulfate and 2% by weight thereof of malt diastase. An analysis of the silage after 20 days was then made and the results are set forth:

| <u>Amount Preservative<br/>Per Ton Silage</u> | <u>pH</u> | <u>Percent<br/>Protein</u> | <u>Vitamin<br/>A</u> | <u>Odor</u> | <u>Color</u> | <u>Texture</u> |
|---|-----------|----------------------------|----------------------|-------------|--------------|----------------|
| A. None                                       | 7.4       | 2.7                        | 0                    | Putrid      | Brown        | Slippery       |
| B. 1/2 lb.                                    | 7.3       | 3.0                        | 0                    | Putrid      | Brown        | Slippery       |
| C. 1 lb.                                      | 7.2       | 4.0                        | +                    | Sl. sour    | Brown        | Normal         |
| D. 2 lb.                                      | 6.4       | 4.7                        | +++                  | Pealike     | Green        | Normal         |

Since fresh peavine silage is 4.7% protein, Test D shows that with 2 lb. per ton of silage, there is full retention of the protein and vitamin A content, while the untreated silage lost almost one-half of its protein and all of its vitamin A. Thus the presence of the minor amount of the preservative of the process has doubled the feeding value of the peavine silage.

When tests were conducted with green oats silage, it was found that the silage which had been treated with 1 1/2 lb. of sodium sulfate containing 2% by weight of malt diastase, retained 82% of its carbohydrates and almost all of its vitamin A content. The untreated

## Forage and Fodder

green oats silage had lost one-half of its carbohydrates and most of its valuable vitamin A content.

Example 2: Treated and untreated baled hay having a moisture content of from 24 to 35% was maintained at a storage temperature of from 88° to 90°F. The treated hay contained one and one-half pounds of sodium sulfate having 2% by weight thereof of malt diastase per ton of hay. Internal temperature of the hay was measured periodically and the results were as follows:

| <u>Time of Storage</u> | <u>Temperature,<br/>°F., Untreated</u> | <u>Temperature<br/>°F., Treated</u> |
|------------------------|--|-------------------------------------|
| 3rd day                | 110 to 112                             | 96 to 98                            |
| 7th day                | 128 to 134                             | 86 to 88                            |
| 14th day               | 144 to 148                             | 84 to 86                            |
| 30th day               | 90 to 92                               | 84 to 86                            |
| Appearance             | Moldy                                  | No Mold                             |
| Odor                   | Musty                                  | Fresh                               |

### Use of Aqueous Ammoniated Superphosphate

T.E. Freese (U.S. Patent 3,457,081; July 22, 1969; assigned to Allied Chemical Corporation) found that corn silage having improved nutritive and keeping characteristics can be obtained by addition of aqueous ammoniated superphosphoric acid to the corn forage prior to ensiling. Preferably, sufficient ammoniated superphosphoric acid is used to provide a silage that will furnish all the phosphorus need of the ruminant. If desired, a small proportion, e.g., up to about 5% by weight, based on the weight of the silage, of calcium carbonate, preferably ground limestone, may be added to the silage. Urea can also be added, if desired, to provide additional nitrogen value.

An aqueous ammoniated superphosphoric acid having the following properties which will hereinafter be referred to as Solution A, was found to be particularly useful.

|  | <u>Percent by Weight</u> |
|--|--------------------------|
| Nitrogen                                       | 10                       |
| Phosphorus (as P <sub>2</sub> O <sub>5</sub> ) | 34                       |
| Trace Minerals                                 | 1 to 2                   |
| Iron (Fe <sub>2</sub> O <sub>3</sub> )         | ca. 1.0                  |
| Calcium (CaO)                                  | ca. 0.1                  |
| Magnesium (MgO)                                | ca. 0.3                  |
| pH   | 6.0                      |
| Specific gravity at 60°F.                      | 1.4                      |
| Salting out temperature °F.                    | 0                        |

Distribution as percent by weight of the ammonium phosphates of Solution A was as indicated in the table on the following page.



|                             | <u>Percent</u> |
|-----------------------------|----------------|
| Ammonium orthophosphate     | 37             |
| Ammonium pyrophosphate      | 49             |
| Ammonium tripolyphosphate   | 8              |
| Ammonium tetrapolyphosphate | 5              |
| Higher ammonium phosphates  | 1              |

Example: Two corn silage treatments were made using Solution A, the aqueous ammoniated superphosphoric acid described above. The forage was a hybrid dent field corn, cut in the early-dent state, and chopped into one-half inch pieces. The moisture value of the forage was about 72%. The treatment plan was to use the Solution A at levels of about 35 and 65 pounds of Solution A per ton of forage. A control silage was also prepared with no Solution A.

The steel silos that were used measured about 12 feet in diameter and 30 feet high. The silos had a holding capacity for 50 to 60 tons of corn silage. In the first silo, the forage was sprayed with Solution A at the rate of 5.6 gallons (about 65 lb.) of Solution A per ton of forage. In the second silo, the forage was sprayed with an equivolume solution of water and Solution A at a rate equivalent to 3 gallons (about 35 lb.) of Solution A per ton of forage. No Solution A was added to the control silage. A Kools blower which had a rated capacity of one ton per minute was used in filling the silos. The Solution A was sprayed on the forage as it was being conveyed into the blower.

After 90 days storage, the silage was evaluated in laboratory and animal feeding tests. Quality of the silage in storage seemed good by appearance and odor. Acidity (defined as milliliter of 0.1 N KOH required to titrate a 9 g. sample in 60 ml. of water to pH 7.0) and pH on the control and two treatments gave the following results:

| <u>Percent Solution A Added</u> | <u>Acidity</u> | <u>pH</u> |
|---------------------------------|----------------|-----------|
| 0                               | 11.8           | 4.0       |
| 1.75                            | 14.7           | 4.1       |
| 3.25                            | 16.1           | 3.9       |

Feeding trials were carried out as follows: Twenty-four 700 to 800 lb. heifers were divided into 3 groups and fed silage containing 0, 1.75 and 3.25% Solution A. About 1% ground limestone was added to both the 1.75% and 3.25% material at feeding time. The animals were observed to feed normally. Blood phosphorus was increased from 6.30 to 7.75 mg./100 ml. by the 1.75% Solution A feed, and from 6.34 to 8.84 mg./100 ml. by the 3.25% Solution A feed. These results demonstrate the availability of the phosphorus to the animals and are considered very favorable. Heifers were also observed to feed normally on 1.75% material to which no limestone had been added.

#### Antioxidant Plus Enzyme Combination

A process developed by T.B. Tribble and E.L. Rondenet (U.S. Patent 3,284,212; November 8, 1966; assigned to Flavor Corporation of America) in which antioxidants are combined with enzymes, is highly effective in preserving silage over extended periods.

Example 1 — Silo 1: An alfalfa hay crop was selected and an untreated portion thereof was ensiled in a first silo under the best known recommended procedures of air, moisture and storage control practice.

Example 2 — Silo 2: A second portion of the same hay crop was also ensiled in a second silo and treated with a recognized commercially available antioxidant preservative formulated as follows:

|                                | <u>Parts by Weight</u> |
|--------------------------------|------------------------|
| Butylated hydroxytoluene (BHT) | 3                      |
| Ethoxyquin                     | 3                      |
| Citric acid                    | 2                      |

Such antioxidant preservative was added to the hay by dusting eight grams thereof over each ton of hay according to recommended and recognized procedure for its utilization.

Example 3 — Silo 3: A third portion of the same hay crop was ensiled in a third silo utilizing a recognized commercial enzyme preservative added according to manufacturers recommended practice and procedures. Such enzyme preservative was compounded as follows:

|           | <u>Parts by Weight</u> |
|-----------|------------------------|
| Diastase  | 6                      |
| Cellulase | 1                      |

This preservative was added to the hay by dusting the same thereover in the ratio of seven grams of enzyme preservative per ton of hay.

Example 4 — Silo 4: A fourth portion of the same hay crop was treated according to the preservation practices of the process with a combined antioxidant and enzyme preservation having the following formulation:

|                                | <u>Parts by Weight</u> |
|--------------------------------|------------------------|
| Diastase                       | 6                      |
| Ethoxyquin                     | 3                      |
| Butylated hydroxytoluene (BHT) | 3                      |
| Citric acid                    | 2                      |
| Cellulase                      | 1                      |

This preservative was added to the hay by dusting substantially 15 g. thereof over each ton of hay which was then ensiled in a fourth silo. After 3 weeks of ensiling, the resulting silages were assayed. The results are given in the table shown on the following page.



## Forage and Fodder

| <u>Assay for</u>    | <u>Silage 1<br/>as is</u> | <u>Silage 2<br/>as is</u>   | <u>Silage 3<br/>as is</u> | <u>Silage 4<br/>as is</u> |
|---------------------|---------------------------|-----------------------------|---------------------------|---------------------------|
| Moisture, %         | 78.12                     | 76.76                       | 74.90                     | 69.10                     |
| Ash, %              | 1.96                      | 2.11                        | 2.07                      | 2.90                      |
| Fat, %              | 0.62                      | 0.80                        | 0.60                      | 0.69                      |
| Protein, %          | 6.68                      | 6.87                        | 7.31 <sub>e</sub>         | 8.85                      |
| Fiber, %            | 4.95                      | 5.18                        | 6.04                      | 3.80                      |
| Carbohy-<br>drate % | 7.67                      | 8.28                        | 9.08                      | 15.37                     |
| pH (acid)           | 5.70                      | 5.20                        | 5.65                      | 4.50                      |
| β-carotene          | 5.60 mg./lb.              | 8.80 mg./lb.                | 7.30 mg./lb.              | 13.00 mg./lb.             |
| Tocopherol          | 1.00 mg./100 g.           | 4.20 mg./100 g.             | 3.60 mg./100 g.           | 2.70 mg./100 g.           |
| Odor                | Acidic/Butyric            | Acidic/Butyric <sup>a</sup> | Acidic/Butyric            | Forage/Lactic             |
| Taste               | Sour/Rancid               | Sour/Rancid                 | Sour/Rancid               | Sweet/Forage/Lactic       |
| Color               | Dark                      | Dark                        | Dark                      | Natural                   |

### Bacterial Fermentation with *Pediococcus Cerevisiae*

W.L. Brown (U.S. Patent 3,147,121; September 1, 1964; assigned to John Morrell & Co.) provides an improved method of ensiling forage material of field and ground plants such as grass, clover, corn, corn fodder, legumes, alfalfa, mixtures thereof and other vegetable and animal forage materials by controlling the bacterial fermentation and completing the bacterial fermentation in a short time by incorporating viable cells of *Pediococcus cerevisiae* in the fodder being ensiled.

The activity of *Pediococcus cerevisiae* can be enhanced by adding a suitable source of carbohydrate assimilable by the microorganism to the green fodder along with the microorganism.

Example: To 1,000 g. of freshly cut grass was added 0.5 g. of *Pediococcus cerevisiae* cells and 9.25 g. of dried molasses. The ingredients were then thoroughly mixed, packed into a glass jar and the covered glass jar allowed to stand at 76°F. for 72 hours. At this time the pH of the silage was found to be 4.1, and a microscopic examination indicated the presence of  $4 \times 10^7$  acid-producing colonies per gram.

In the same way 1,000 g. of the same cut grass was packed into a jar, the jar covered and allowed to stand at 76°F. for 72 hours. At the end of this time, the pH was found to be 5.0, and a microscopic examination indicated the presence of  $6 \times 10^7$  of nonacid-producing microorganisms per gram.

### Streptomycin Plus Streptomycin Resistant Lactic Acid Bacteria

In a process developed by S. Hashimoto (U.S. Patent 3,459,554; August 5, 1969; assigned to Kaken Kagaku Kabushiki Kaisha, Japan) the ripening of silage is improved by admixing with the green silage an antibiotic substance, a lactic acid bacteria culture resistant to the added antibiotic and an accelerator for the lactic acid fermentation.

**Example — Prescription of Addition Agents (per Ton of the Raw Materials):** As an anti-microbial substance, 5 ppm of streptomycin is employed. As lactic acid bacteria having resistance against the antimicrobial substance, one billion of *St. lactis* (the suitable temperature is 10°C.; 1% of lactic acid is formed at a temperature ranging from 10° to 38°C.) having 1,000γ/cc of resistance against streptomycin and one billion of *L. plantarum* (the suitable temperature is 25°C.; 1.3 to 1.5% of lactic acid is produced at a temperature ranging from 10° to 40°C.) are used.

As general lactic acid bacteria, the same amount of the above mentioned two kinds of lactic acid bacteria, which have no resistance is used. As an accelerator of increasing lactic acid bacteria, 200 g. of powdered skim milk is used.

**Siloes for the Tests, Raw Materials and a Process for Feeding Materials:** As test siloes, 6 specimen bottles having 12 cm. in diameter and 39 cm. in depth are well washed with water and dried. As the raw materials, Chinese milk vetches are picked immediately before they are in full bloom, exposed slightly to the sun, cut to fine pieces and stirred. Thereafter, the prepared raw materials are fed in each of siloes with pressing. The top of a thermometer, in the form of a rod, is then placed in the center of the silage. The scale rod of the thermometer is thrust out of the silo through the center of the cap so that the temperature of the silo can be observed. The whole contents are strongly pressed and the bottle is closed by the cap. Then, each of the bottles is placed in a cold place in a storehouse. The temperature of the outside world is 20° to 22°C., and the temperature in the storehouse is 18°C.

TABLE 1: RESULTS IN TEST SILOES

| Silo Number.....   | No. 1      | No. 2                      | No. 3                             | No. 4  | No. 5   | No. 6   |
|--|------------|----------------------------|-----------------------------------|--|---|---|
|  |            |                            |                                   |  |   | An anti-microbial substance, lactic acid bacteria having resistance and an accelerator of increasing lactic acid bacteria |
|  | Comparison | Lactic bacteria acid alone | An anti-microbial substance alone | An anti-microbial substance and accelerator of increasing lactic acid bacteria | An anti-microbial substance, lactic acid bacteria and an accelerator of increasing lactic acid bacteria |   |
| Tests up to stabilization:   |            |                            |                                   |  |   |   |
| Days until the highest temperature appears during fermentation.....              | 4          | 3.5                        | 3                                 | 3  | 2.5   | 2   |
| The highest temperature, °C.....   | 36         | 33                         | 30                                | 29   | 27  | 24  |
| Days required until silages are stabilized.....                                  | 31         | 30                         | 29                                | 28   | 25  | 22  |
| Test after stabilization:  |            |                            |                                   |  |   |   |
| pH.....  | 4.3        | 4.2                        | 4.7                               | 4.6  | 4.4   | 3.9   |
| Dried materials, percent.....  | 20         | 19                         | 20                                | 20   | 19  | 19  |
| Partial loss, <sup>1</sup> percent.....  | 7          | 5                          | 3                                 | 2  | 2   | 1   |
| NH <sub>4</sub> -N, <sup>2</sup> percent.....                                    | 3.0        | 2.5                        | 1.6                               | 1.3  | 1.0   | 0.5   |
| Lactic acid, <sup>2</sup> percent.....   | 7          | 8                          | 2                                 | 3  | 7   | 13  |
| Butyric acid, <sup>2</sup> percent.....  | 1          | 0.8                        | 0.5                               | 0.3  | 0.2   | 0   |
| Acetic acid, <sup>2</sup> percent.....   | 3.0        | 2.2                        | 1.8                               | 1.8  | 1.8   | 0.5   |
| Propionic acid, formic acid, succinic acid and others, <sup>2</sup> percent..... | 1.6        | 1.5                        | 1.2                               | 1.2  | 1.1   | 0.6   |
| Whole organic acids, <sup>2</sup> percent.....                                   | 12.6       | 12.5                       | 5.5                               | 6.3  | 10.1  | 14.3  |
| All anaerobic bacteria <sup>3</sup> .....  | 200,000    | 250,000                    | 3,000                             | 8,000  | 20,000  | 350,000   |
| Lactic acid bacteria <sup>3</sup> .....  | 120,000    | 260,000                    | 2,000                             | 5,000  | 15,000  | 345,000   |
| Butyric acid bacteria <sup>3</sup> .....   | 1,000      | 500                        | <1                                | <1   | <1  | 0   |

<sup>1</sup> Percent, based on the total amount of the silages.

<sup>2</sup> Percent, based on the amount of solid materials.

<sup>3</sup> Number of bacteria in 10 g. of the silages; the unit is 1000.

In addition silage prepared according to this process is superior to all others in smell, taste, color and feel.



## Zinc Bacitracin Plus Molasses

A method of preserving silage with zinc bacitracin has been developed by F.N. Andrews (U.S. Patent 2,940,857; June 14, 1960; assigned to Purdue Research Foundation). The zinc bacitracin can be used in combination with the nutritive preservatives of the character previously used, such as molasses, corn or other suitable form of carbohydrate.

Two pound samples of material were placed in plastic bags as described by Walker et al (J. Animal Science, 13, 1013). The forage was a mixture of Brome grass and alfalfa in the 1/4 to 1/2 bloom stage. Molasses was added at levels of 40 and 80 pounds per ton of silage. pH determinations were made at different intervals and are recorded in Table 1.

TABLE 1

| Treatment                           | pH,<br>1 Month | pH,<br>3 Months | pH,<br>6 Months |
|-------------------------------------|----------------|-----------------|-----------------|
| <b>Bacitracin:</b>                  |                |                 |                 |
| 0.....                              | 4.1            | 4.4             | 4.8             |
| 4 gm./ton.....                      | 4.0            | 4.1             | 4.9             |
| 20 gm./ton.....                     | 3.9            | 4.1             | 5.0             |
| 40 gm./ton.....                     | 4.6            | 4.0             | 4.8             |
| <b>Bacitracin+40 Lbs. Molasses:</b> |                |                 |                 |
| 0.....                              | 4.5            | 4.2             | 4.6             |
| 4 gm./ton.....                      | 3.7            | 4.6             | 4.6             |
| 20 gm./ton.....                     | 4.3            | 4.4             | 4.2             |
| 40 gm./ton.....                     | 4.5            | 4.3             | 3.8             |
| <b>Bacitracin+80 Lbs. Molasses:</b> |                |                 |                 |
| 0.....                              | 3.8            | 4.4             | 4.1             |
| 4 gm./ton.....                      | 4.0            | 4.3             | 4.0             |
| 20 gm./ton.....                     | 4.2            | 4.6             | 3.5             |
| 40 gm./ton.....                     | 3.6            | 4.2             | 3.8             |

Table 2 below shows the chemical analyses of the products at the end of the 3 months for the products shown in Table 1.

TABLE 2

| Treatment                           | Dry Matter, Per-cent | Protein, Per-cent | Ether Extract, Per-cent | Fiber, Per-cent | N Free Extract, Per-cent | Ash, Per-cent |
|-------------------------------------|----------------------|-------------------|-------------------------|-----------------|--------------------------|---------------|
| <b>Bacitracin:</b>                  |                      |                   |                         |                 |                          |               |
| 0.....                              | 14.17                | 4.82              | 0.94                    | 4.39            | 13.23                    | 1.83          |
| 4 gm./ton.....                      | 13.15                | 4.71              | 1.22                    | 4.48            | 11.93                    | 1.57          |
| 20 gm./ton.....                     | 12.54                | 4.36              | 1.07                    | 4.21            | 11.47                    | 1.48          |
| 40 gm./ton.....                     | 13.28                | 4.87              | 0.87                    | 4.43            | 12.41                    | 1.78          |
| <b>Bacitracin+40 lbs. Molasses:</b> |                      |                   |                         |                 |                          |               |
| 0.....                              | 14.60                | 4.50              | 0.77                    | 4.55            | 13.83                    | 1.86          |
| 4 gm./ton.....                      | 15.30                | 4.69              | 1.26                    | 4.52            | 14.04                    | 1.84          |
| 20 gm./ton.....                     | 15.22                | 4.57              | 0.94                    | 4.13            | 14.28                    | 1.80          |
| 40 gm./ton.....                     | 15.80                | 4.79              | 0.97                    | 4.99            | 14.83                    | 1.93          |
| <b>Bacitracin+80 lbs. molasses:</b> |                      |                   |                         |                 |                          |               |
| 0.....                              | 13.99                | 4.48              | 0.92                    | 4.63            | 13.67                    | 1.83          |
| 4 gm./ton.....                      | 15.35                | 4.52              | 1.07                    | 4.42            | 14.28                    | 2.01          |
| 20 gm./ton.....                     | 17.75                | 4.59              | 1.01                    | 4.40            | 16.74                    | 1.83          |
| 40 gm./ton.....                     | 17.37                | 4.72              | 1.42                    | 5.10            | 15.95                    | 2.03          |

The presence of zinc bacitracin in silage not only does not prevent a highly satisfactory fermentation, but also preserves the nutrients present when molasses also is used.

## Forage and Fodder

### Sodium Tripolyphosphate and Sodium Hexametaphosphate

A process for preserving silage by adding sodium tripolyphosphate or hexametaphosphate has been developed by J.E. Thompson (U.S. Patent 3,368,901; February 13, 1968). Corn for green ensilage is usually harvested about the time that its grains start to form a depression on the top of the kernel. The ensilage includes the stalk of the corn, the fodder and the entire ear of corn. This is usually chopped and blown into the top of the silo. At the time the ensilage is blown in, the sodium salts of hexametaphosphate or sodium tripolyphosphate are added (1% or 20 lb./ton). The material is allowed to settle for 1 or 2 days and then the vacant space at the top of the silo is refilled with green chopped ensilage containing the additive.

Example: After a period of approximately one year comparative siloes of untreated and treated material gave the following results:

| Test<br>100 Ton<br>Silo | Untreated                |                          |                          | Treated                  |                          |                          |
|-------------------------|--------------------------|--------------------------|--------------------------|--------------------------|--------------------------|--------------------------|
|                         | Wet<br>Basis,<br>Percent | Dry<br>Basis,<br>Percent | Lb./Ton<br>Green<br>Corn | Wet<br>Basis,<br>Percent | Dry<br>Basis,<br>Percent | Lb./Ton<br>Green<br>Corn |
| Protein.....            | 3.24                     | 13.50                    | 49.05                    | 3.52                     | 11.56                    | 60.33                    |
| Fat.....                | .89                      | 3.72                     | 13.47                    | 1.13                     | 3.72                     | 19.37                    |
| Fiber.....              | 3.20                     | 13.35                    | 48.45                    | 4.07                     | 13.35                    | 69.76                    |
| NFE*                    | 15.01                    | 62.55                    | 227.25                   | 18.90                    | 62.02                    | 323.95                   |
| Mineral.....            | 1.65                     | 6.88                     | 24.98                    | 2.85                     | 9.35                     | 48.85                    |
| Total Dry.....          | 24.00                    | 100.00                   | 363.20                   | 30.48                    | 100.00                   | 522.26                   |
| Total Moist.....        | 76.00                    | -----                    | 1,150.65                 | 69.52                    | -----                    | 1,191.57                 |
| All                     | 100.00                   | -----                    | 1,513.84<br>=75.7%       | 100.00                   | -----                    | 1,713.83<br>=85.7%       |

\*When a feed is analyzed for water, protein, fat, fibre and mineral the total of these is subtracted from 100 to give a percentage known as "NFE".

The use of sodium salts of tripolyphosphates and hexametaphosphates apparently form a gel and retain the proteins and carbohydrates within the silo and prevent the usual drainage which accompanies fermentation within the silo.

### Addition of Dry Substances Containing Starch and Protein

In the process of H. Biehl (U.S. Patent 3,172,764; March 9, 1965) a method is provided for the production of a fermentation fodder in which a mixture is produced comprising fresh fodder, dry substances containing starch and dry substances containing protein and is stored in a silo. Where the fresh fodder contains less than about 2% of sugar the mixture also includes dry substances containing sugar.

Example: The table shown on the following page shows the results obtained with tests when storing in a silo a sugar-containing green fodder and a green fodder practically free of sugar. The values shown under (a) were obtained without the addition of dry components with juice drainage, the values under (b) were obtained without the addition of dry components and without juice drainage, and those under (c) were obtained with storage in a silo in accordance with the process. For any one type of fresh substance to be ensilaged, a correspondingly formulated mixture of dry components and auxiliary substances is made up and added in a ratio of about 75% of fresh substance and 25% of dry components. These components are



## Forage and Fodder

then intimately mixed when stored in the silo, either in a separate mixing process or directly while being charged into the silo while preserving the requisite mixing ratio.

| Green fodder              |  | Mixture of dry components   |                         | Loss of dry mass, percent | Acidic condition |                      |                      |                       | Utilization of the fodder by animals  |
|---------------------------|--|---|-------------------------|---------------------------|------------------|----------------------|----------------------|-----------------------|---|
| Kind                      | Amount of material stored in the silo, percent | Composition   | Amount in silo, percent |                           | pH-value         | Lactic acid, percent | Acetic acid, percent | Butyric acid, percent |   |
| Beets of nutritive value. | -----  | 80% tapioca-meal.<br>20% rough-ground soya.<br>40g./ton aureomycin.                   | -----                   | 46                        | 4.1              | 0.4                  | 0.7                  | -----                 | 10% better than with (a) and (b) calculated on the percentage of beets in the fodder.                           |
|                           | (a) 100  | -----   | -----                   | 25                        | 4.0              | 0.16                 | 1.0                  | -----                 |   |
|                           | (b) 100  | -----   | 25                      | 5                         | 3.85             | 1.7                  | 0.25                 | -----                 |   |
| Grass                     | -----  | 78% tapioca-meal.<br>16% dates.<br>6% rough-ground soya meal.<br>40g./ton aureomycin. | -----                   | 10.5                      | 5.1              | 0.4                  | 0.7                  | -----                 | About 20% better than with (a) and (b) or with fresh grass calculated on the percentage of grass in the fodder. |
|                           | (a) 100  | -----   | -----                   | 6.1                       | 4.8              | 0.55                 | 0.65                 | 0.01                  |   |
|                           | (b) 100  | -----   | 25                      | 3.2                       | 3.65             | 1.85                 | 0.2                  | -----                 |   |

### Fermented Feed Substance Without Green Fodder

Another process of H. Biehl (U.S. Patent 3,246,989; April 19, 1966) provides for the production of improved feed substances by fermentation, without the addition of green fodder, which comprises making a dry comminuted mixture of cereal grain, a highly digestible protein containing feed ingredient and supplements, converting the mixture into a pulpy mass by admixing 54.5 to 240% of water by weight for each 100% by weight of the mixture having a usual water content of about 15% by weight, putting the pulpy mass into a silo and ensilaging it.

During ensiling, lactic acid fermentation will occur in the known manner with air being precluded and with no juice being extracted during the ensiling process. After a period of about 4 to 6 weeks withdrawing may begin. The fermented feed forms a rather thick pulpy layer. This mass may be taken without any further treatment from the lower part of the silo and may then be directly distributed amongst the animals by means of a feeding cart.

The processing of the mixed grain may be produced by means of a mobile mixing plant which consists of a water container for the metered addition of the water, a container for withdrawing the ready mixture, and a pump below the container serving to deliver the mixture into the silo.

Example 1: A suitable feed for calves, which can be ensilaged by this process is shown on the following page.

## Forage and Fodder

|   | <u>Percent</u> |
|---|----------------|
| Linseed meal or extracted coarse-ground grain (linseed)                               | 30             |
| Coarse-ground grain (barley, corn)  | 20             |
| Oil cake or extracted coarse-ground oil seed (e.g. extracted coarse-ground soy beans) | 15             |
| Oatmeal   | 10             |
| Dried skim milk   | 10             |
| Sweet whey powder   | 5              |
| Cereal milling by-products (e.g. wheat bran)  | 5              |
| Dried yeast   | 3              |
| Mixtures of mineral substances with vitamins and antibiotics                          | 2              |
|   | <u>100</u>     |

### Example 2 — Utilization of the Nonsilaged and the Silaged Feed by the Animal:

| Group No. | Kind of feed | Number of animals | Initial weight in kilograms | Final weight in kilograms | Total gain in weight per animal in kilograms | Daily gain in weight per animal in grams | Total feed consumption in kilograms dry mass per animal | Kilograms dry mass fed to obtain 1 kilogram of living weight gain | Weight gain per total feed fed, percent |
|-----------|--------------|-------------------|-----------------------------|---------------------------|--|--|---|---|---|
| 1         | Not silaged  | 10                | 74.000                      | 84.625                    | 10.625                                       | 759                                      | 29.273  | 2.76  | 26.3                                    |
|           | Silaged      | 10                | 73.000                      | 84.250                    | 11.250                                       | 804                                      | 28.782  | 2.56  | 39.1                                    |
| 2         | Not silaged  | 10                | 70.000                      | 80.000                    | 10.000                                       | 732                                      | 28.069  | 2.74  | 36.5                                    |
|           | Silaged      | 10                | 71.250                      | 83.125                    | 11.875                                       | 848                                      | 28.454  | 2.40  | 41.7                                    |

It has furthermore been observed that hogs which have been fed on silaged feed have a better flesh to fat ratio, which shows that the protein is better utilized by the animal after fermenting.

### Storage of Grain Under Carbon Dioxide

The process of P.E. Fayhee (U.S. Patent 2,952,541; September 13, 1960) relates to crop storage and preserving, and more particularly to structures and methods for storing and preserving grains and legumes.

A storage container, bin or silo may be positioned on the ground or on any suitable foundation. The storage bin is sealed and is substantially air tight and may be constructed out of steel, for example. The bin is adapted to contain grain, such as corn, ensilage, silage or legumes, and these may be withdrawn from the bin for use by means of a door located at the bottom of the bin.

A bottle gas container (external source) having carbon dioxide under pressure therein is connected by a pipe or tubing with the storage bin at the top of the bin. A pressure regulator of conventional construction is in the pipe and regulates the carbon dioxide that flows through



the pipe to the top of the bin between zero and 10 lb. per square inch. In operation, the bin or silo is filled or partially filled with grain, ensilage, corn, silage or legumes by conventional methods, such as from the top through any suitable trap door. With the door being closed, the bin is then very nearly air tight. The regulator or other valve in the line is then opened, and the regulator then allows carbon dioxide to fill the bin and the regulator maintains the carbon dioxide pressure in the bin between zero and 10 lb. per square inch.

The carbon dioxide, being heavier than air, passes downwardly from the top of the bin and forces the air up where it remains at the top of the structure away from the stored material, and the bin contents are thus completely surrounded by carbon dioxide. When contents of the bin are removed for usage through the door and the tank is again connected through the pipe with the top of the bin after having been disconnected when the contents were being removed, the carbon dioxide again moves downwardly within the bin and fills the bin.

The application of carbon dioxide from the tank to the bin advantageously prevents the usual fermentation of the bin contents that occurs, which has some limited preserving functions, and preserves the bin contents for a longer time in more nearly its original condition.

### DRYING OF FORAGE MATERIAL

For centuries man has been concerned with the drying of cut forage crops. It is well known that the faster the drying process can be undertaken, the less likelihood of degradation. It is also been known that certain drying techniques have deleterious effects and reduce the nutritional quality of the stored crops. Some progress has been made in recent years in the preservation of the nutritional qualities of the forage crops by improved handling and storing techniques not only in the drying but also in the ensiling of the forage. Nevertheless, with present methods, large nutritional losses still occur, and drying times are still lengthy.

#### Fast Forage Drying Technique

An improved fast forage drying method is described by E.H. Hess (U.S. Patent 3,218,172; November 16, 1965; assigned to Sperry Rand Corporation). When a plant is severed from its root system, the stomata quickly close in an effort to preserve life of the individual cells as long as possible, thus encapsulating the water and making it difficult to remove. Over the remaining plant surface there exists a waxy coating which serves as a barrier against moisture loss.

This process is aimed at maintaining the stomata in open position to accelerate drying by either altering or removing the waxy leaf and stem coating which acts as a moisture barrier.

Techniques: Steam Treatment — Samples were suspended over boiling water for 2 to 5 minutes. Solvent Dipping Experiments — Samples were dipped for 30 seconds in chloroform, benzene or perchloroethylene. Solvent Vapor Treatments — Samples were exposed to boiling vapors of perchloroethylene (BP 121.2°C.) and chloroform (BP 61.2°C.) for 2 to 5 minutes. Air Drying Experiment — The above treated samples were placed on the floor of a well ventilated room in which the temperature did not exceed 30°C.

The drying rates of alfalfa subjected to solvent dipping and solvent vapor treatment were compared with those of steamed and untreated material. It should be noted that the sample which was exposed to hot perchloroethylene vapors reached the 20% moisture level in 1/3 of the time required for the steamed sample and 1/7 of the time required for the control. It is also significant to note that each treated sample retained at least 4% more total dry matter than the control. The samples from the air drying experiment were submitted to thorough chemical analysis.

TABLE 1: ANALYSIS OF AIR-DRY

|   | Control | Choro-<br>form<br>dip | Perchloro-<br>ethylene<br>dip | Benzene<br>dip | Chloro-<br>form<br>vapor | Perchloro-<br>ethylene<br>vapor | Steam<br>(2 min.) | Steam<br>(5 min.) |
|---|---------|-----------------------|-------------------------------|----------------|--------------------------|---------------------------------|-------------------|-------------------|
| Percent Ash (dry wt. basis).....                      | 7.30    | 6.82                  | 7.04                          | 6.70           | 7.02                     | 6.80                            | 6.96              | 6.73              |
| Dry wt. in fresh wt. conversion factor.....           | .222    | .237                  | .230                          | .239           | .231                     | .238                            | .233              | .241              |
| Percent increase in dry matter retention.....         | 0       | 6.75                  | 3.60                          | 7.65           | 4.05                     | 7.20                            | 4.95              | 8.55              |
| Protein content (as percent of fresh wt.).....        | 4.99    | 5.26                  | 5.06                          | 5.33           | 5.17                     | 5.24                            | 5.17              | 5.25              |
| Percent enrichment over control.....                  | 0       | 5.41                  | 1.40                          | 6.81           | 3.60                     | 5.01                            | 3.60              | 5.21              |
| Crude fiber content (as percent of fresh<br>wt.)..... | 6.53    | 6.90                  | 6.62                          | 6.72           | 6.65                     | 6.64                            | 6.52              | 7.08              |
| Carotene content (IU/lb. based on fresh<br>wt.).....  | 10,500  | 6,070                 | 7,540                         | 7,410          | 8,960                    | 38,100                          | 29,800            | 42,900            |
| Percent change in carotene content.....               | 0       | -42.2                 | -28.2                         | -29.4          | -14.7                    | +263                            | +184              | +308              |
| Estimated TDN (as percent of fresh wt.).....          | 14.6    | 15.6                  | 15.2                          | 16.0           | 15.3                     | 16.0                            | 15.6              | 15.8              |
| Percent increase in TDN.....                          | 0       | 6.84                  | 4.10                          | 9.58           | 4.79                     | 9.58                            | 6.84              | 8.21              |

It was concluded that the experimental treatments especially the chloroform and benzene dips and perchloroethylene vapor treatment are capable of effecting considerable improvements in the dry matter retention and nutritional value of alfalfa.

Effect of Heat Treatment — Hot Air: Two milliliter quantities of perchloroethylene and 5% IGEAL (a non-ionic surfactant) were sprayed onto alfalfa. The baskets were placed in a forced-draft oven for 2 minutes at 185°C. then removed and cooled to room temperature. These heat treated stalks were placed in the oven at 60°C. for 30 minutes. The percent total moisture removed is given below.

|                          |       |
|--------------------------|-------|
| Untreated master control | 52.6% |
| Heat treated control     | 63.7% |
| IGEAL                    | 72.7% |
| Perchloroethylene        | 80.6% |

Ironing Technique: Another more practical method of applying heat in a field operation might be passing the hay between hot rollers. In an effort to reduce this technique to a laboratory scale, heat was applied for 30 seconds to alfalfa with an ordinary household iron. The percent total moisture removed is given below.

|                   |       |
|-------------------|-------|
| Master control    | 45.2% |
| Control           | 69.5% |
| Perchloroethylene | 88.6% |
| IGEAL             | 83.2% |

The ironing technique in combination with the chemicals produce the most dramatic increases



in drying rate of the methods tested.

Chemical Additives: Because the cost of many solvents for large scale operation might be prohibitive, a series of surface active agents and related materials were tested. In this survey aqueous solutions of various additives were sprayed onto alfalfa in 2 ml. portions. The alfalfa was then heat treated according to the ironing technique already described.

| Ironing time | Material                       | Description   | Percent sol. | Percent weight loss through 30 mins. drying at 60° C. |
|--------------|--------------------------------|---|--------------|---|
| 30 secs.     | Control                        | Control   |              | 69.0  |
|              | Orzan "S"                      | Sodium salt of sulfonate lignin (An emulsion stabilizer). | 5            | 71.2  |
|              |                                |   | 20           | 80.6  |
|              | Alkyl benzene sulfonate (ABS). | Anionic detergent   | 2            | 80.0  |
|              | IGEPAL                         | Non-ionic surfactant                                      | 2            | 83.2  |
|              | Triton X-200                   | Anionic surfactant  | 2            | 80.2  |
|              | Triton X-400                   | Cationic surfactant                                       | 2            | 81.6  |

### Increasing Protein Content of Forage Material

J.F. Simonet and C.P. Castle (U.S. Patent 3,063,839; November 13, 1962) found that when freshly harvested plant material is treated with a nitrogenous material, preferably urea, and then subjected to heat, an enhancement of the naturally occurring proteinaceous content of the plant material is obtained.

The permissible elapsed time between cutting and further processing varies in accordance with the particular atmospheric conditions existing, and generally will not exceed 8 hours. The main conditions are that when further processing takes place, the green forage plant material should not have any substantial discoloration or browning and should still have a fresh odor.

The heating mechanism is adapted to operate at atmospheric pressure, the heating means being controlled to provide a temperature of the green forage plant material within the dehydrating tube that lies within the range of 160°F. to an upper limit of 220°F. The upper limit combined with the time of heat treatment has been chosen to prevent degradation of the end product. The separated material is treated within the aforementioned temperature range for a period of at least one half minute. The moisture content of the green forage plant material is reduced during the heating process to less than approximately 20% moisture, and preferably in the range of 5 to 13%, whereby it is apparent that a major portion of the moisture content of the plant material is effectively removed during the treatment thereof.

Example: Alfalfa was separated from its root system in the field and transported to the point of processing. At the time of arrival of the separated material at the process equipment, the alfalfa had been separated from the plant less than 8 hours, was not discolored and had a fresh odor. At the process point, the surfaces of the alfalfa were substantially wetted by spraying the surfaces with a solution of urea and water just prior to entering the heat treatment zone. The wetted alfalfa was conveyed through the heat treatment zone where the

## Forage and Fodder

temperature of the product was raised to approximately 192°F. and maintained at this level for 1/2 minute so that the end product had an average moisture content of about 8.7%. A control sample was provided for comparison. 26,020 lb. of alfalfa having an initial average moisture content of 80% was treated with 923 lb. of urea and water solution containing 246 lb. of moisture-free urea (4.73% urea to weight of alfalfa). The following table illustrates the increase of the various substances.

|                          | Grams per 100 g.<br>(Moisture-Free<br>Basis) — Control<br>Material | Grams per 100 g.<br>(Moisture-Free<br>Basis) — Example | % Increase<br>(Moisture-Free<br>Basis)— Example<br>over Control |
|--------------------------|--|--|---|
| Crude Protein (N x 6.25) | 14.79  | 24.31  | 64.5  |
| Methionine               | 0.39   | 0.65   | 67  |
| Leucine                  | 0.7  | 1.8  | 157   |
| Lysine                   | 0.7  | 1.2  | 72  |
| Isoleucine               | 1.3  | 1.3  | ±0  |
| Phenylalanine            | 1.3  | 1.1  | -15   |

The larger the percentage of urea to alfalfa employed, the greater the enhancement of the gross crude protein. It has been found that an upper limit of 10% of urea to alfalfa by weight on a moisture-free basis is critical and an increase of the percentage of urea to alfalfa beyond this point produces unsatisfactory results.

The alfalfa product of this process has a carotene content superior to that of comparable dehydrated alfalfa products and is provided with a noticeably greener color. Furthermore, the scorched odor which is present in commercially dehydrated alfalfa is completely avoided and the odor of the end product is sweet and fresh.

### Reducing Carotene Loss During Storage

Drying of crops in the field or in drying equipment results in a loss of valuable food accessory factors. An important requirement of a satisfactory inhibitor is that it is substantially nonvolatile under the conditions of use so that it will not be evaporated and lost during the drying treatment.

J.A. Chenicek (U.S. Patent 2,944,903; July 12, 1960; assigned to Universal Oil Products Company) has developed a process for stabilizing carotene-containing crops subject to oxidative deterioration which comprises spraying the crops with N,N'-di-alkyl-p-phenylene diamine, where the alkyl group contains 8 or 9 carbons.

The spraying or dusting of the crops may be done while the crops are in the field, either prior to or after cutting, or the inhibitor may be applied after the crops have been gathered and stored. The amount of inhibitor to be applied to the crops in general will range from 0.001 to 0.5% or more by weight of active ingredient.

Example 1: Alfalfa containing 28 mg. of carotene per 100 g. will lose approximately 50% of its carotene content when stored at 75°F. for 7 weeks. However, upon the addition of



N,N'-di-(1-ethyl-3-methylamyl)-p-phenylene diamine dissolved in soy bean oil, in an amount of 0.25% by weight based on active inhibitor compound, the carotene content of the alfalfa is considerably higher when stored at 75°F. for 7 weeks.

Example 2: When the alfalfa is stored at 125°F. for 7 weeks, it will lose approximately 77% of its carotene value. Here again, the addition of 0.25% by weight of the active inhibitor described in Example 1 reduces the loss of the carotene content.

### Preserving Hay

A process for preserving hay which comprises encasing a compressed mass of hay in a substantially air tight container, and treating the hay to inhibit the growth and reproduction of bacteria, spores and mold has been developed by J.A. Haase (U.S. Patent 3,050,396; August 21, 1962).

An optimum container for use in the process is a bag of flexible, tough synthetic sheet material which is substantially fluid-impermeable and which may be sealed at its ends against substantial ingress or egress of fluid such as clear polyethylene sheet material in gauges within the range between 0.050" and 0.100". The hay is treated in such a fashion as to reduce its moisture content to a value within the range between 20 and 50% before packing it into the containers. Such moisture reduction can be accomplished by physical squeezing or by heating or by a combination of those two actions.

Sterilization can be achieved, before compression and packaging, by flash-heating hay by passing it, in a relatively thin layer, over a highly heated drum, holding the hay in contact with the drum long enough so that every particle of the hay will attain a temperature of approximately 180°F. for at least 10 seconds. Alternatively, it has been found that, if the hay is compressed and packaged in air-tight containers, of the character above described, without preliminary heating, and then is allowed to lie in the field, under a summer sun, for a period of at least two bright days, temperatures in the range between 120° and 150°F. will be attained within the package and the hay will keep satisfactorily for indefinite periods, presumably because activity of the detrimental organisms has been inhibited by such long exposure to such temperatures.

Example: Exhaustive tests were run upon hay which had been flash heated to 180°F., treated for reduction of moisture content and then compressed and bagged and stored for a period of approximately 6 months, with the following results:

Moisture: 53%.

pH (acidity as hydrogen ion concentration) 5.0. This is believed to indicate the formation of some lactic acid.

Molds: Very few.

Bacteria: Gram positive types present, some in fair numbers. No gram negative found. The gram positives included:

- (a) Some spores of spore forming bacteria. Of course, these are not killed in any pasteurizing treatment.
- (b) Some inert cocci. These are rather heat resistant and akin to those

## Forage and Fodder

causing high counts in some pasteurized milk.

- (c) Cocci and a few rods which had reproduced slightly (as in silage, only not as much) giving rise to the lactic acid found.
- (d) Some actinomycetes. Probably streptomyces. These are undesirable—if they were to reproduce much they would give an earthy odor as in freshly spaded garden soil. Slight reproduction would not be noticed by man or (presumably) cow.

### Meal from Forage Plants

J. Branom (U.S. Patent 2,949,362; August 16, 1960; assigned to Standard Alfalfa Milling Company) has developed a method for producing a meal for animal feeding purposes, from forage plants, such as alfalfa, and from hay, and the like, which meal is highly palatable and nutritious and may comprise as in the case of alfalfa, discrete leaf sections substantially free of dust or fine particles and having coarseness and bulk for necessary roughage.

Alfalfa immediately upon being cut is permitted to be sun dried or cured in the field for a requisite period of time. The cured or dried alfalfa is then steamed for moisture absorption to assure the absorption of sufficient moisture by the cured alfalfa for softening the leaves thereof, or in other words, to produce wilting.

The now steamed, moisture-laden alfalfa, the temperature of which has, understandably, been raised above atmospheric temperature, is then conveyed from the steam compartment to a conventional rotary type chopper. Because of its steamed condition, the alfalfa will not, under the forceful strokes of the chopper, be reduced to powdered or dusty form by such slicing action, but will merely be divided into discrete leaf portions, which may be approximately of the order of  $3/4$  of an inch, constituting the meal.

From the chopper the steamed alfalfa meal is transmitted, by suitable means, to a drying unit to remove the moisture or steam from the meal, which would include the applied steam as well as any that may have been generated from water originally contained in the alfalfa upon subjection to the elevated temperatures in the steam compartment. By traverse through the dryer the alfalfa meal is rendered substantially fully dried, although remaining at a relatively high temperature.

At this time the meal is ready for discharge into a downwardly directed cooling conduit or pipe which is thin walled and atmospherically surrounded, for gravity-motivated movement therealong. Flowing counter-wise to the direction of movement of the alfalfa meal is a cooling-air current of such force relative to the gravitational pull of the alfalfa meal as not to impede the even descent of the meal.

Also, the current is at such a relatively reduced temperature as to permit heat exchange therebetween, so that upon arrival of the meal at the lower end of the cooling conduit the temperature of the meal will have been lowered to substantially atmospheric temperature. From the conduit the alfalfa meal is collected for packaging, as in bags, or other related devices.



## OTHER FODDERS

### Fodder from Chopped Straw

A process for producing fodder from straw by the addition of yeast to a bath containing straw, water and an alkaline inorganic compound after such a mixture has been heated to a sufficient extent so as to partially cause degradation of the lignin and pentosan structures within the cell walls of the straw and heating the mixture containing this yeast until plasmolysis of the yeast is obtained has been developed by A. Flechsig (U.S. Patent 2,940,858; June 14, 1960; assigned one-half to Trans-Oceanic).

Example: One hundred pounds of finely divided or chopped straw was placed in a wooden tank containing steam tubes in approximately three equal layers; 1.6 lb. of sodium hydroxide were positioned between each of the three layers of the straw. The tank was then heated for a period of one hour at 212°F. through the use of steam supplied through the tubing. At the end of this period 60 lb. of water was added and the mixture was boiled for another hour.

After this, the entire reaction mixture was cooled. After it had reached a temperature of 40°C. a lime-yeast mixture containing 10 lb. of quick lime, 25 lb. of water and 3 lb. of dry beer yeast was added to the entire reaction mixture and the resulting mixture was stirred. An additional 25 lb. of water was then added. The entire mixture created as a result of these operations was then cooked through the use of steam at 212°F. for a period of two hours. After this was done, the reaction solution or mixture was neutralized with hydrochloric acid to a pH of about 7.

In general, about one cubic centimeter of concentrated hydrochloric acid is required per kilogram of reaction solution in order to affect neutralization. The so neutralized mixture was then dried to a moisture content of about 20 to 23% by heating it at a temperature of from 212° to 250°F.

### Silica Substitute for Roughage in Feed

A feed ration in which a substantial portion of amorphous hydrated silica of predetermined size and shape characteristic substitutes for the conventional feed roughages is described by J.H. Baker (U.S. Patent 3,244,527; April 5, 1966).

The amorphous hydrated silica has a sieve analysis of 100% through a U.S. #10 sieve; 30% retained on a U.S. #30 sieve; 60% retained on a U.S. #40 sieve; and 100% retained on a U.S. #50 sieve. This is a high-grade, white silica which is hard, rounded and smooth-surfaced with an American Foundry Society grain fineness of 27, as from the Ottawa district of Illinois. If the silica is coarser than the above range it will not be accepted by cattle; if the silica is smaller in size it is not effective.

The following formula is fed at the rate of 1 lb. per day of this formula with 9 lb. of milo per 400 lb. of animal weight; i.e., 200 lb. of this formula is mixed with 1,800 lb. of milo to form each ton of total feed mixture; and 10 lb. of the resultant total mixture is fed per 400 lb. of animal weight, in the form of pellets or mash with molasses as binder.

TABLE 1: CONCENTRATE AND SILICA RATION FORMULA

| <u>Item</u>  | <u>Weight</u>                |
|--|------------------------------|
| Vitamin D <sub>2</sub> (80,000,000 int'l units)                          | 1 lb.                        |
| Vitamin K  | 1 lb.                        |
| Vitamin A250 (113,400,000 U.S.P. units)                                  | 1 lb.                        |
| Copper sulfate   | 1/4 lb.                      |
| Flavor   | 1/2 lb.                      |
| Iodine   | 60 g.                        |
| Urea   | 240 lb.                      |
| SiO <sub>2</sub>   | 633 lb.                      |
| Dicalcium phosphate  | 180 lb.                      |
| Yeast  | 21 lb.                       |
| Salt   | 100 lb.                      |
| Magnesium sulfate  | 3 lb.                        |
| Choline  | 1 lb.                        |
| KCl  | 20 lb.                       |
| Fat  | 20 lb.                       |
| Stilbosol  | 10 lb.                       |
| Sodium sulfate   | 50 lb.                       |
| Riboflavin   | 240 g.                       |
| Cottonseed meal (or soybean meal)  | 710 lb.                      |
| d-Pantothenic acid (as dl-calcium pantothenate calcium chloride complex) | 13,900 mg.                   |
| Niacin   | 15,000 mg.                   |
| Choline chloride   | 37,500 mg.                   |
| Vitamin A (as vitamin A palmitate)                                       | 60,000,000 U.S.P. units      |
| Vitamin D <sub>2</sub> (as d-activated plant sterol)                     | 30,000,000 Int'l chick units |
| Vitamin E (alpha tocopheryl acetate)                                     | 45,000 Int'l units           |
| Manganese (as sulfate)   | 15 g.                        |
| Cobalt (as carbonate)  | 1.7 g.                       |
| Copper (as carbonate)  | 1.7 g.                       |
| Zinc (as oxide)  | 10 g.                        |

Example: A group of 60 steer calves and yearlings of good market grade, average age 8 to 9 months, average weight of 355 lb., were randomly assigned to six lots of 10 animals each. 10 animals (Lot 5) were slaughtered at the beginning of the trial to represent initial body composition and condition. The other four groups were assigned to different treatments. At the time of slaughter of the initial groups, all animals were changed to their respective assigned rations and the experimental feeding period started.

The action of the silica appears to be not only on the bulk or volumetric characteristics of the feed but also on the in vivo metabolism of the carbohydrate. A very large amount of propionic and butyric acid relative to acetic acid is found in the rumen of the animals fed the silica-containing ration.



TABLE 2: ANIMAL TESTS

|               | Lot #1  | Lot #2   | Lot #3                                | Lot #4                                 | Lot #5     | Lot #6                                 |
|---------------|---|--|---------------------------------------|--|------------|--|
| Days Fed:     |   |  |                                       |  | Slaughter. |  |
| 20            | SiO <sub>2</sub> +<br>Conc.<br>Ration.<br>Slaughter | SiO <sub>2</sub> +<br>Conc.<br>Ration.           | Conc.<br>Ration<br>only.<br>Slaughter | Conc.<br>Ration<br>only.<br>(1)        |            | SiO <sub>2</sub> +<br>Conc.<br>Ration. |
| 134           |   | Conc.<br>Ration<br>without<br>SiO <sub>2</sub> . |                                       | SiO <sub>2</sub> +<br>Conc.<br>Ration. |            |  |
| Liver Scars   | No  | Yes  | Yes                                   | Yes                                    | No         | Slaughter.<br>No.                      |
| Liver Abscess | No  | Yes  | Yes                                   | No                                     | No         | No.                                    |
| Carcass Grade | Choice  |  |                                       |  |            | Choice.                                |

(1) Foundered and off feed.

The animals are also treated with a water solution of ethylene diamine dihydro iodide and of water-soluble vitamin A dissolved in a drinking tank. The tank holds 500 gallons of water and is provided with 60 g. of ethylene diamine dihydro iodide and 45,000,000 units of vitamin A. This procedure provides for drinking and absorption by the animals of the supplemental vitamin A needed, notwithstanding the absence of legumes and the high acid concentration of the ruminant stomach during intake of the high energy ration.

The volume of feed consumed is substantially less by this process than by conventional methods. The average daily gain using the composition of Table 1 exceeded that of the conventional feeding. A composition for use in feeding cattle is prepared by mixing 12 lb. of animal fat per 100 lb. of silica. The resultant mixture of animal fat and silica is readily accepted by feed cattle due to the flavor of the animal fat and is eaten independently of or with regular rations to provide up to 3% by weight of the total weight of feed consumed by each animal.

### Hydroponically Produced Vegetal Fodder

A process developed by A. Malchair (U.S. Patent 3,131,064; April 28, 1964; assigned to Bureau d'Etudes Armand Malchair SA, Belgium) for producing vegetal compositions substantially consists in spreading to a convenient thickness the material which is to form the permanent support; in spreading the grains or seeds over the latter, without any special preparation; and in saturating periodically aforesaid permanent support and the aforesaid grains or seeds with an aqueous medium containing nutrient substances.

When the plants have reached a certain stage of development, it will be preferable to expose the vegetal composition which has thus been obtained to the solar radiation, so as to let it grow further exactly as if the vegetation were growing in normal soil. The vegetal composition substantially consists of a permanent support of a growth the roots of which become embedded in the aforesaid permanent support as is the case with plants grown in normal soil.

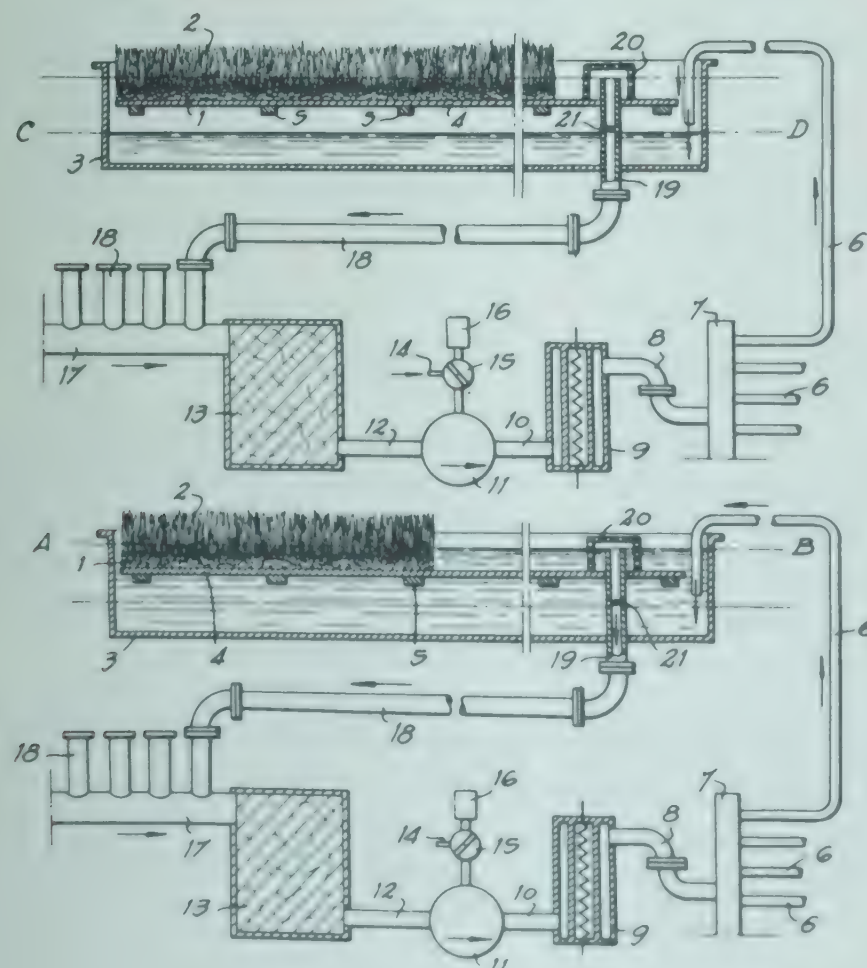
In the particular case when it is intended to grow fodder, the permanent support 1 will preferably be made of a comparatively thin bed of dry grass, which initially will of course be

fairly loose but, once the vegetal composition has been formed, this permanent support will acquire a certain compactness and a fairly good resistance on account of the large amounts of roots and rootlets entwined therein. It will be observed, that this mass is in itself an actual fodder without any waste nor substances which can not be assimilated by the animals.

An apparatus for the production of a new vegetal composition by hydroculture is illustrated in Figure 2.1. It comprises at least one fairly shallow trough 3 the longitudinal and transverse dimensions of which can be chosen ad libitum; at a certain distance above the bottom of this trough, there is a false bottom 4 supported for instance by battens 5; one of the tubes 6 connected to a manifold 7 dips into the aforesaid trough 3, the manifold being connected by a tube 8 to a heat exchanger 9.

The input of the latter is connected to a pump 11 the input of which is connected to a container 13 designed so as to act as a filter. The aforesaid pump 11 comprises an air inlet 14 controlled by a motor driven valve 15. The movements of the latter, as well as the electric circuit of the driving motor (not represented) of pump 11 are controlled by a clockwork 16.

FIGURE 2.1: HYDROPONICALLY PRODUCED VEGETAL FODDER





A manifold 17, to which all return pipes 18 are connected, opens into the upper part of the aforesaid container 13. The other end of each of these return pipes carries a tubular element penetrating through the bottom and the false bottom 4 of trough 3 and extending upward so as to reach a level A—B which is close to the upper face of the permanent support 1 onto which the grains or seeds have been spread. The upper open end of the aforesaid tubular element 19 is provided with a strainer 20 and the wall of this tubular element has a series of small holes 21 provided at the level C—D reached by the liquid during the heating phase of the operating cycle.

The operating cycle which characterizes the method takes the following course: considering that a bed of dried grass constituting the permanent support has been spread into the false bottom 4, and that over this bed a layer of grains or seeds has been spread, the protection of these grains or seeds, of the germs and of the roots and rootlets against dangerous variations of temperature is secured by putting into circulation a liquid medium, which generally is water to which nutrient substances have been added, through the circuit comprising the filtering container 13, pipe 12, pump 11, pipe 10, the heat exchanger 9, pipe 8, manifold 7, the corresponding pipe 6, that part of trough 3 which is comprised below the level C—D, the openings 21 of the tubular element 19, the return pipe 18 and the manifold 17.

In order to maintain the regulating liquid at the maximum level C—D in the trough 3, the air inlet 14 is opened to the required extent, which so to say regulates the rate of delivery of the pump. The water-air mixture is forced at reduced rate into the trough 3, whereby the openings 21 of the tubular element act as an overflow. The returning liquid is filtered in container 13, whilst the temperature of the water-air mixture can be properly regulated by means of some known form of thermostat or aquastat inserted into the circuit and controlling the switching-on and off of the resistance heaters of the heat exchanger 9. Under these circumstances, whatever the ambient temperature may be, the permanent support 1 as well as the grains or seeds, the roots and rootlets will always be maintained at an optimum temperature and will at any rate be protected against all dangerous variations of temperature.

For the purpose of saturating periodically with the nutrient solution aforesaid permanent support and the elements which it contains, all that is necessary is to include into the program of the operating cycle a periodic closing of the motor driven valve 15 so as to close the air inlet 14 of pump 11. The rate at which the latter will be discharging water thereby increases and the level of the liquid medium in trough 3 will rise, considering that the openings 21 provided in the tubular element 19 cannot cope with this increased rate of flow. The liquid thus reaches the level A—B which is approximately at the same height as the upper face of the permanent support 1 of the vegetal composition.

The excess of liquid medium flows away through the top of the tubular element 19 which thus acts as a constant level device. Finally, when the soaking has lasted long enough, the motor driven valve 15 is put once more into action through the clockwork device 16 and opens in a duly controlled way the air inlet 14 of the pump. The rate of discharge of the latter will thus diminish once more and the liquid medium will therefore recede below the false bottom 4 until it reaches the level C—D, i.e., the level of the heat regulation phase.

The new technique will produce actual blocks of fodder made up of a perfectly edible permanent support combined with whole grass without any alteration, such blocks lending



themselves to be preserved in a perfect state of freshness during exceptionally long periods, due to the presence of the nutrient solution permeating the support as well as the roots. The result of the foregoing is that the grass continues to live normally and may even grow further when stored, without impairing its nutrient properties. A first surprising consequence of this method is to be found in the fact that the plants need only stay for a very short time in the actual installation, as their further development can take place in the same way as in soil cultivation, either under natural light or artificial light radiation.

Another surprising consequence of this new technique is to be found in the fact that the aforesaid blocks can almost at once be cut up into separate rations of predetermined weight, each ration being fed to the animals in the same way as a ration of fresh fodder.

### FORAGE PELLETS

The general process of making a pelletized feed consists in mixing the feed ingredients for example alfalfa hay chopped to suitable sizes with a suitable binder and molding or extruding the mixture to produce a pellet or briquette the size of which depends on the ultimate use. The use of pelletized feeds has been widely adopted and is becoming increasingly popular because of convenience in handling as well as an actual saving in the feed consumed by preventing waste and losses when loose feeds, or feeds in meal form are used in the feeding trough. It is necessary that the pellets be compact and firm and resistant to attrition during handling.

#### Enzyme Treated Starch Binder for Forage Pellets

The process developed by E.H. Hess and D.R. Tshudy (U.S. Patent 3,420,671; January 7, 1969; assigned to Lancaster Laboratories, Inc.) consists in converting high carbohydrate cereal grains or other vegetable matter in such a manner (enzymatic hydrolysis) that they develop adhesive qualities as pellet binders not previously possessed by them.

The amount of the hydrolytic product to be mixed with the chopped alfalfa or other forage material or animal feed is preferably about 1% to about 3% on a dry basis, depending on the type of binder and other factors. After mixing, the resulting material is subjected to sufficient pressure to compress the mixture into durable pellets or briquettes preferably upward of 1,000 psi to several thousand psi, employing conventional equipment of the extrusion, piston or rotary types to carry out this step of the process.

Example 1 — Preparation of Binding Agents: (A) Slurry Form — The grain, which may consist of corn, barley, oats, wheat and the like or a mixture thereof, was crushed and ground, e.g., in a hammermill equipped with a fine mesh screen. The finely divided grain was then mixed with water in this example in the proportion of one part grain to three parts water, and about one-tenth of 1% by weight of a commercial amylolytic enzyme preparation (e.g. of the type described as liquifying diastase of bacterial origin, Rohm and Haas Rhozyme H-39) added and the mixture was then allowed to incubate for about 2 hours at a temperature of about 45°C. (Since commercial enzyme preparations vary widely in their diastatic activity, the amount of enzyme necessary to effect the grain conversion will also vary.) The mixture was then transferred to a pressure cooker, and cooked for about 15 minutes at a steam pressure of about



15 psig (121°C.). The product thus produced could be mixed with about three parts of water by weight or sufficient to produce a slurry which could be easily mixed with chopped alfalfa or other forage product or crushed grain.

The binder product as described above is usually in the form of a heavy syrup containing very small suspended particles and may be used directly as a binder (or mixed with water as stated) through the liquid injection system usually found in pellet mills. Alternatively, if desired, or found necessary, the product of the processed cereal grain may be dried and reduced to a fine powder prior to use. Preferably the drying operation in such cases should be of the type where the product is not exposed to high temperatures for long periods, such as treatment in processes of the spray dryer or vacuum type evaporators.

(B) Damp digestion-laboratory equipment —Finely ground grain prepared as described in the previous example was dampened with water in the ratio of 2 parts to 1 part water. Pre-dissolved in the water used to dampen the grain was an amount of commercial enzyme preparation (same as previously described) equal to one-tenth of one percent based on the weight of grain used. This mixture, contained in a beaker, was placed in a boiling water bath for 30 minutes during which time its internal temperature rose to 70° to 75°C. The beaker was then transferred to a pressure cooker where it was treated at 15 psig (121°C.) for 15 minutes. Part of the product was allowed to air dry to about 10% moisture content (later referred to as dry binder 1) and to the remainder was added sufficient water to provide a 1 part grain to 4 parts water ratio (later referred to as slurry binder 2).

Example 2 — Evaluation of Hydrolytic Products as Pellets Binders: (A) Sets of pellets were prepared from low moisture alfalfa (about 8.3% moisture) and amounts of the pellet binders prepared as described in Example 1A were mixed with chopped alfalfa, so that the binders contributed 1.5% grain solids and 9.0% water to the mixture. These pellets were prepared in a hand operated press which had been standardized to give equivalent results to the commercially operated pelletizing machines. A series of tests employing various grains was made using shelled corn, whole corn (cob included), barley, oats and wheat. Control pellets were similarly prepared using 9% of water instead of the binders, prepared however under the same conditions of time, temperature and pressure.

The comparative quality of the pellets was evaluated by subjecting sets of six pellets to the physical action of a standardized washing machine type agitator for a period of 30 minutes followed by determination of particle size distribution by weight measurement. Particle size distribution was reduced by a standardized scoring system to a single number falling between 0 and 500. The results obtained are shown below.

| <u>Grain</u>              | <u>Quality Index</u> | <u>Density lb./ft.<sup>3</sup></u> |
|---------------------------|----------------------|------------------------------------|
| Control                   | 313                  | 32.0                               |
| Shelled corn              | 405                  | 32.7                               |
| Whole corn (cob included) | 412                  | 33.3                               |
| Barley                    | 408                  | 32.8                               |
| Oats                      | 412                  | 31.6                               |
| Wheat                     | 402                  | 31.2                               |

## Forage and Fodder

(B) Hydrolyzed grains prepared according to the process of Example 1B, dry binder 1, were ground to a fine powder and tested after storage. One part of air dried processed grain was stirred for a short time with seven parts of lukewarm water (60°C.) and this slurry was used as a binder for pellets from a low moisture hay. The results are shown below.

| <u>Grain</u> | <u>Quality Index</u> | <u>Density lb./ft.<sup>3</sup></u> |
|--------------|----------------------|------------------------------------|
| Control      | 194                  | 33.7                               |
| Shelled corn | 323                  | 35.2                               |
| Barley       | 372                  | 37.9                               |

They clearly demonstrate that, if desired, the binder slurry can be dehydrated for purposes of storage or transit, then rehydrated at the time of use with no loss of binding qualities.

### Wafer from Forage Material

A method of producing and a product made from forage crops to be used as livestock feed whereby forage materials are compressed or condensed into a wafer, briquette or biscuit which preferably can be from 3 to 4 inches in diameter and three-quarters to 1 inch thick, are described by W.C. Briggs and F.W. Hoover, Jr. (U.S. Patent 2,995,445; August 8, 1961; assigned to Sumner Iron Works, Inc.).

The method of producing a wafer product from forage crops comprises preparing the forage material. The preparation includes harvesting the material and if necessary increasing or decreasing the moisture content to within the range of 12 to 25%. The material is then chopped or cut into lengths of from 1 to 3 inches. This is accomplished by conventional grain or grass chopping equipment. The material is then conveyed to a hopper and from the hopper it is fed into a compression machine. The particular construction or method of operation of the machine is not the subject matter of this process. Any machine which will produce the desired results may be employed.

The material is continuously fed to the machine by gravity flow from a hopper. The machine compresses each charge into a wafer approximately one inch thick. The diameter of the wafer may vary but for reasons previously stated, it is preferred to make a wafer 3 to 4 inches in diameter. The charges of material are condensed or compressed to a hardness or density whereby the product has a specific gravity within the range of 0.9 to 1.1. This causes the material to be bonded together so that it will readily adhere and not disintegrate when handled or stored.

There are certain critical requirements in the process or method of producing a satisfactory wafer product. For example, the temperature must be regulated from 75° to 125°F. so as to limit the glazing and removal of moisture. It is also necessary to produce a relatively cold wafer and to control the temperature of the parts of the machine. The wafer after being formed in a compression chamber is then passed through a holding or extruding chamber where it is held under substantial pressure. It is not satisfactory to merely form the wafer in a compression chamber and immediately discharge it from the machine. A wafer product composed of forage crops in combination with nutritional supplements can also be manufactured which will be in the form of a complete food.



Feed Pellet of Unchopped Hay

A process for making a feed article of unchopped hay of such dimensions as to be readily acceptably by the animal in its mouth in one piece for mastication, and provide the body of the pellet with an interior space freely and permanently opened to the direct access of air has been developed by J. Molitorisz (U.S. Patent 3,357,835; December 12, 1967; assigned to Michigan State University).

The process is described with reference to Figure 2.2a. Hay 25 of the length originally mowed is continuously fed from a hopper 26 (used merely as symbolic of a means of continuous feeding) down to the uppermost of a series of rolls 27 which surround equidistantly, a rapidly rotating spindle 28. These rolls may be provided with fixed axes and complementary bearings but as shown they are yieldingly borne at one end at least toward the spindle 12 by springs 29 suitably connecting with their axes 30 and biasing the axes through radial slots 31 in movement limiting suitably fixed members (not shown) radially inward toward the spindle 12. Rotations being in the directions shown the hay fed between the upper rolls is borne toward the rapidly rotated spindle 12 and is wrapped about it.

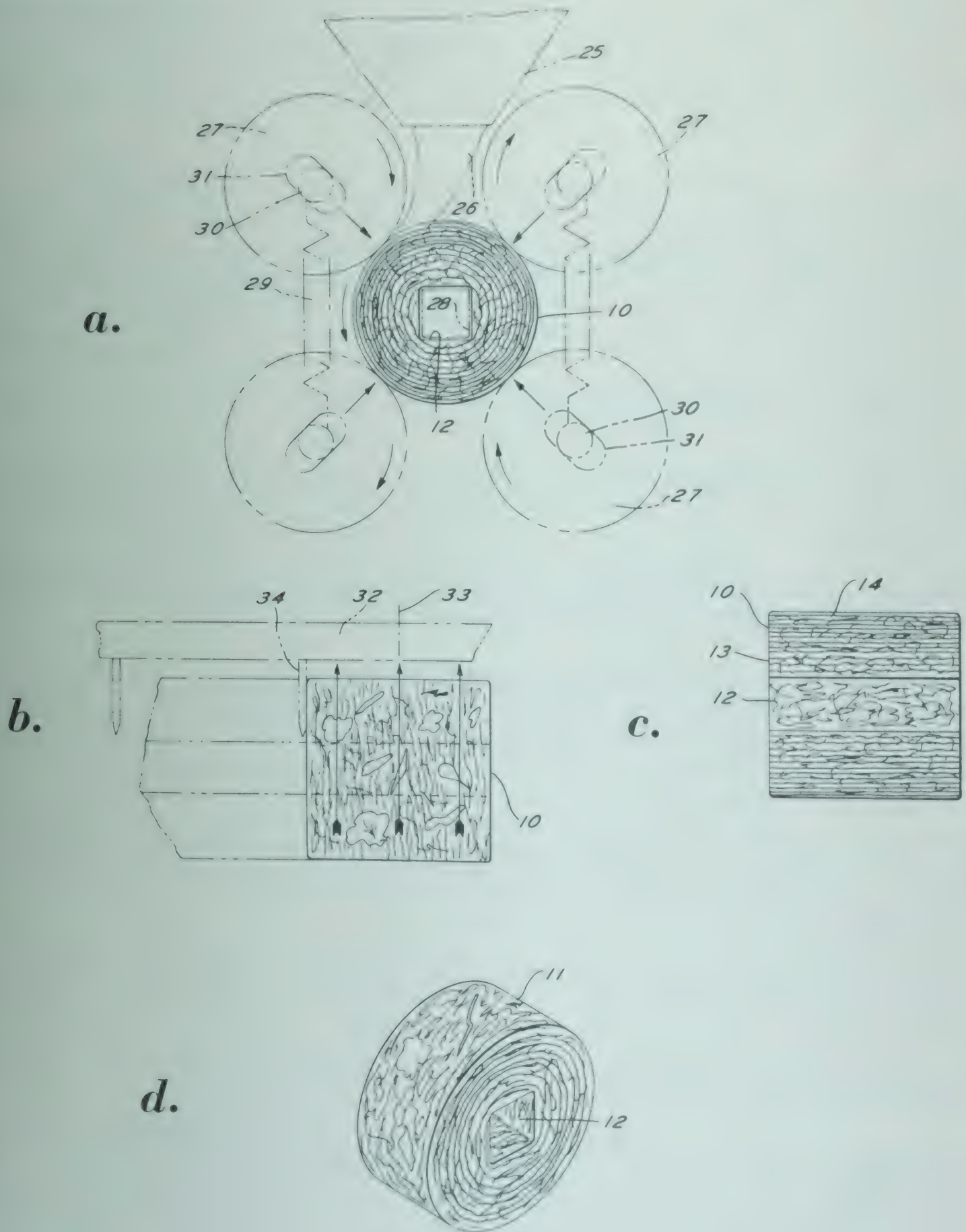
Upon its reaching a wrapped diameter contacting the rolls 27 the spirals of hay from this point onward are subjected to very considerable compression due to the springs which bear the rolls radially against it, and finally to the fact that the axes 30 of the rolls bottom in the slots 31 and the pressure is further heightened. If the axes be fixed in position all the compression results from such fixation. The innermost layers are less tightly wrapped because initial compression is absent, and pressure at that time results from wrapping only.

The outermost layers are the more tightly wrapped because the pressure of the rolls is added to the pressure of wrapping. As compression progresses the outer layers are moved radially inward and condensing, crushing, breaking and interlocking results. This takes place outstandingly in the outer layers but also in the inner layers. Too the outer layers are subjected to pressure for the longer time. Bonding juices are exuded from the crushed portions. As so completed the articles are pushed off the spindle (by means not shown). When pushed off the spindle 12 the height of pressure exerted on the innermost layers is relieved to a degree by reason of the manufactured opening 12, though the opening per se is retained substantially of full dimension.

Thus, through the use of these means (and others not portrayed) the density of certain of the inner portions 13 is made appreciably less than that of the outer portions 14. For good handling conditions he prefers an average inner portion density of approximately 20 lb. per cubic foot and an average outer density of approximately 30 lb. per cubic foot, at 15 to 18% moisture or an equivalent ratio of densities of inner to outer portions 2 to 3.

The material from which these articles are manufactured may be of a wide range of moistures, anything from 15% upwards to the neighborhood of 80 to 90% and within this range they can be dried satisfactorily. Illustrated in phantom in Figure 2.2b the knife means 34 is carried by a bar 32 for severing the wrapped material into the desired axial portions. The knife means are shown inserted into cutting position by lowering the bar 32 while spindle 28 is still rotating but after hay feeding has ceased. Obviously, knife means 34 may have any desired spacing to form articles of different lengths. When the axial length (or thickness) is about

FIGURE 2.2: FEED PELLET OF UNCHOPPED HAY





1 inch or 1 1/4 inches it is all the more readily acceptably receivable in the mouth of the animal for mastication. With the axial dimension reduced the diametral dimension may be acceptably larger.

Figure 2.2c is an axial cross section of the feed article of Figure 2.2a. Figure 2.2d is a prospective view of a feed article of less axial dimension.

### Pelleting Hay at Low Pressures

The process of W.N. King (U.S. Patent 3,013,880; December 19, 1961; assigned to FMC Corporation) provides an improved method of preconditioning hay having a moisture content of between 14 to 35% prior to forming the same into pellets. In practicing the process, hay having a moisture content between 14 to 35% is gathered from the field by the pelleting machine thereby making it possible to shorten the time required to dry the hay to pelleting condition by several days.

The hay is then masticated, and during the masticating operation, the hay is crushed, and torn, ruptured or cut into random lengths usually of about one-half inch or less. Occasionally a few longer lengths are present and these long lengths aid in providing an interlaced pellet structure. The mastication causes the breakdown of the natural fibrous structure of the stems resulting in the liberation of the glutenous juices in the stems and in the leaves which act as a binder when the masticated hay is pressed into pellets. Also, during this step, the natural resilience of the stems is destroyed and the resulting product is in the form of an inelastic, damp mass of limp material which has very little tendency to expand after it has been formed into pellets.

This masticated material is then fed into dies and is compressed into pellets of any suitable shape and size. A press having a two inch diameter bore has been used successfully to form pellets of commercial size which are two inches in diameter and are between one-half and one inch in length. The pellets are formed by using pressure in the range of 200 to 1,600 psi depending upon the moisture content of the masticated material and the desired density of finished pellet. It has been found that pellets formed by this method and having a density of 25 lb. per cubic foot or higher will maintain their pellet shapes when subjected to normal handling techniques. Pellets having densities up to 45 lb. per cubic foot, can be formed by pressures in the above range.

Pellets having either a low or a high density can be formed by this process at much lower pressures, and hence lower energy values than are required when forming pellets from chopped or ground hay. For example, when forming a pellet having a density of 30 lb. per cubic foot from masticated hay, only 1/9 as much pelleting energy is required as is required for making pellets from chopped hay. It is believed that this energy saving is due to the removal of the natural resilience of the hay during the masticating step.

After the pellets have been released from the forming dies, they are transported to storage bins, or the like, and are allowed to dry in order to prevent the formation of mold. Under normal atmospheric conditions, no special drying techniques other than adequate ventilation are required. The outer surfaces of the dried pellet have a somewhat glazed appearance and, when the pellet is broken to reveal the internal structure, it is noted that many hairlike fibers

## Forage and Fodder

and flat stem pieces are interwoven to provide a relatively strong pellet even though its density is close to the lower end of the range of desirable densities. The color and odor of the pellets resemble the color and odor of the same type of hay when baled at rather high moisture contents. The following table covers the characteristics of 12 test pellets formed following this process. These commercial size test pellets are 2 inches in diameter and between 1/2 inch to 1 inch in length, and their expansion after release of pressure and during the drying period did not cause a change in length in excess of 1/16 of an inch.

| <u>Pellet No.</u> | <u>Pelleting Pressure, psi</u> | <u>Moisture Content When Pelleted (Wet Basis) %</u> | <u>Density of Dried Pellets, lb./ft.<sup>3</sup></u> |
|-------------------|--------------------------------|---|--|
| 1                 | 200                            | 20  | 28.2   |
| 2                 | 400                            | 20  | 31.0   |
| 3                 | 800                            | 20  | 40.6   |
| 4                 | 1,600                          | 20  | 45.6   |
| 5                 | 200                            | 29.6  | 32.8   |
| 6                 | 400                            | 29.6  | 35.6   |
| 7                 | 800                            | 29.6  | 41.3   |
| 8                 | 1,600                          | 29.6  | 37.5   |
| 9                 | 200                            | 34.2  | 24.8   |
| 10                | 400                            | 34.2  | 24.4   |
| 11                | 800                            | 34.2  | 26.0   |
| 12                | 1,600                          | 34.2  | 29.4   |

The density of test pellets 1 to 4 was taken after a drying period of more than one week and the density of pellets 4 to 12 was taken after a drying period of more than one month. All of these pellets have a final moisture content of between 10 and 15%.

### Pelleting Roughage Crops

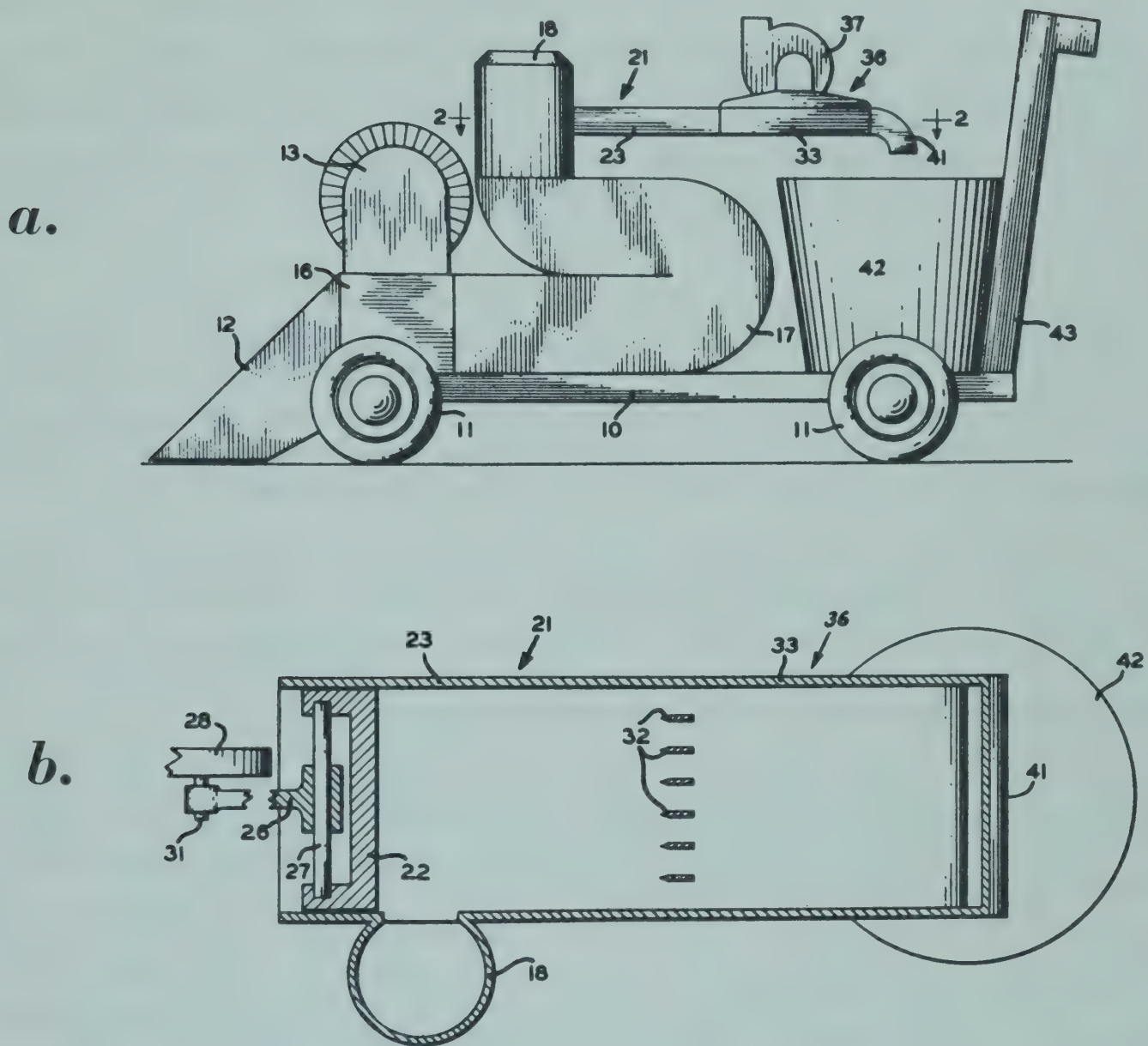
M.A. Kosch (U.S. Patent 2,942,976; June 28, 1960; assigned to Kosch Co.) provides a process by which roughage material such as hay in an unground state may be formed into pellets under low pressure. The hay is compressed at temperatures above 160°F. into a desired shape, and kept in the desired shape during cooling thereof.

Referring to Figure 2.3a, a transit type of apparatus having a base 10 with wheels 11 rotatably secured thereto. A conventional cutter pickup or pickup head 12 extends downwardly from one end of the apparatus and the apparatus may be drawn leftwardly so that a crop may be moved and picked up. Figure 2.3b is a schematic section along line 2-2 of Figure 2.3a.

A conventional burner or gas turbine is indicated by the numeral 13 and is shown mounted above a conventional chopper or hammer mill 16 which is secured to the base 10. The hay passes from the cutter or pickup head 12 into the chopper or hammer mill 16 where the hay is reduced to short lengths before dehydration in a dehydrator 17. The dehydrator 17 with the associated burner or gas turbine 13 should be capable of reducing the moisture content of the hay from around 70% by weight (as in green hay) to a value in the range of from 5 to 30% by weight. The dehydrator 17 heats the hay to a temperature somewhere above



FIGURE 2.3: PELLETING ROUGHAGE CROPS



Source: M.A. Kosch; U.S. Patent 2,942,976; June 28, 1960

approximately 160°F. but not greater than approximately 212°F. or the boiling temperature of water. A certain amount of moisture should remain in the partially dried hay; 5 to 30% by weight seems to give the best adhesion of the hay in the final pelleted product. The thus heated hay passes out of the dehydrator 17 into a conventional hay-air separator 18, which may be mounted upon the dehydrator 17. The hay is charged from the hay-air separator 18 into a compacting means, indicated generally by the numeral 21. This charging operation may be accomplished in a manner similar to the charging of a conventional baler and may be accomplished in cycles, that is, a piston 22 rams the hay rightwardly as viewed in Figure 2.3b and is then retracted. Hay is then charged into the leftward end of a conduit 23, a portion of which forms a part of the compacting means 21. This cycle is repeated again and again causing compacted hay to move rightwardly in the conduit 23.

The heating of the hay to temperatures above 160°F., in the dehydrating step as above described causes the hay to be reduced to a plastic consistency readily allowing the large fibers of hay to quickly conform and maintain the irregular patterns required in the compacted final pelleted product. By heating the hay to temperatures in the range of 160° to 200°F., the pressures used in the compacting step need only be as great as approximately 100 psi to 300 psi instead of the much higher 4,000 psi normally required in previously used pelleting operations. In the case of pressures close to 100 psi, somewhat higher temperatures around 200°F. are necessary to give a properly compacted product. When temperatures close to 160°F. are used, pressures close to or above 300 psi are necessary to give a properly compacted product.

The piston 22 should produce a pressure of 100 to 300 psi at the end of its ram stroke. In the case of the apparatus disclosed herein, however, pressures in the range of 300 to 500 psi are necessary for the following reason: a plurality of knives 32—32 are spaced transversely across and secured to the upper and lower surfaces of the conduit 23 with their cutting edges projecting toward the entrance end of the conduit 23. The added force required to force the compacted hay past these knives makes necessary the additional ram pressure.

Apparatus for accomplishing this process will operate satisfactorily if the dimensions between the knives are from 1 to 2 inches and the vertical inside dimension of the compacting portion of the conduit 23 is approximately 1 inch. The amount of hay which is charged into the conduit 23 before each ramming operation is of a sufficient amount that, when compacted, its dimension longitudinally of the conduit 23, is approximately one inch.

The section 33 of the conduit 23 which is beyond the knives 32—32 is a portion of a cooling means, indicated generally by the numeral 36 because within the section 33 the compressed material is cooled. The section 33 is so proportioned and arranged that the pressure created within the hay by the compacting means 21 is partially maintained so that the pressure within the hay passing through this section is approximately 50 psi. Suitable means such as conventional springs exerting spring pressure upon the sides of the section 33, which sides could be made movable transversely of the direction of movement of the hay, might be incorporated in the device to restrict the passage of hay through section 33 so as to maintain pressure therein.

Referring to Figure 2.3a, the cooling means 36 comprises the section 33 of the conduit 23 and also a cooling air blower 37 secured upwardly of the section 33 of the conduit 23. Even though the hot compacted hay being forced rightwardly through the section 33 is quite dense, it is sufficiently porous to allow the cooling air blower 37 to suck air through the hay causing it to be cooled at least to a temperature of approximately 115°F. before it reaches a curved deflector portion 41 at the far rightward end of the conduit 23.

It has been found that about 2 lb. per square inch of pressure drop acting for a period of one minute is sufficient to cool the hay down to the 115°F. temperature. It should be noted that the heated hay plank passing out of the compacting means 21 should be maintained in the shape which it has as it exits from the compactor 21 until it has been cooled at least to a temperature of approximately 115°F. because not until it reaches this temperature does the compressed shape of the hay become firm enough to withstand handling. In section 33 of conduit 23, a track with rollers or any suitable means may be provided for maintaining it in



this shape until sufficient cooling has been accomplished. When the thus formed plant of hay reaches the curved deflector portion 41 of the conduit 23, it will be forced against the portion 41 and broken along the planes of least resistance which are the planes extending transversely across the plank of compressed hay between the original charges of hay. As the compressed hay drops from the conduit 23 into a carrying bin 42 mounted upon the base 10, it separates along the partitions produced by the knives 32-32 and breaks along the planes between the original charges of hay producing a plurality of compressed firm pellets having a density somewhere in the range of approximately 40 to approximately 60 lb. per cubic foot and having rectangular shapes with dimensions of one inch by one inch by two inches. An unloading elevator 43 may be provided for moving the pellets out of the carrying bin 42 into a truck or storage means.

### Pelleted Feed Containing Fresh Green Forage

A. Lent (U.S. Patent 3,044,877; July 17, 1962; assigned to Erly-Fat Livestock Feed Co.) has developed a process to provide a pelleted animal feed which contains a relatively high content of green forage juices and cells and which can be stored for relatively long periods of time without the formation of mold. This comprises pelleting fresh green forage with cellulosic waste material (cotton gin waste, bagasse, pineapple waste etc. which are capable of absorbing 30% or more liquid). In the preparation of green forage for pelleting, it is advisable to grind the forage as finely as possible, as by means of a hammermill or chopper.

The greater the rupturing of the cells, the more the grass juice is released and the easier the chopped green forage and its juice will mix and absorb in other materials with which it is pelleted. When the young green forage is added to the cellulosic waste material and the mixture is compressed into pellets using pressures of from 10,000 to 60,000 lb. per square inch, the waste material provides the bulk and the envelope or casing means for protecting the forage juices and cells and the green forage provides the nutrients and moisture content which make the entire unit palatable.

In the formation of pellets the use of the fresh green forage reduced the forming temperatures to approximately one half with a resultant reduction in the destruction of the nutrient values (vitamin A, etc.). Pelleted feeds containing 10 to 33% fresh green forage (of 65 to 85% moisture) can be prepared by this process.

The amount of green forage which can be used in the pellet in the absence of mold inhibitors is limited primarily by the amount of resultant moisture in the pellet. Thus, 14% moisture in the resulting pellet does not present any molding problem, and 19% moisture will not mold in dry areas.

However, when mold inhibitors are used, pellets containing as high as 31% moisture did not mold during an approximately 3 months' period. Sodium propionate 0.003% is one of the most efficient inhibitors of mold in this type of product. Therefore, if it is desirable to have pellets or cakes with higher than about 14% moisture, this can be achieved by the use of this inhibitor which can be mixed with the feed material before pelleting, either in the dry form or dissolved in water or molasses.



## FEED LICKS AND BLOCKS

Licks are normally placed at various points in fields where they are readily accessible to the animals. They are provided to enable animals to make up deficiencies of diet in natural grazing or to obtain nutrient, prophylactic, or therapeutic substances. These licks are of necessity exposed to some extent to the weather and since they contain water-soluble substances, they are normally particularly vulnerable to the action of rain and dew, consequently there has hitherto been considerable risk of loss by dissolution. It is an object of these processes to reduce this risk of loss without at the same time either impairing the nutritive value or reducing the availability of the lick to an animal.

### Chalk-Glucose Licks

R.C. Harris (U.S. Patent 3,087,819; April 30, 1963; assigned to Corn Products Company) has a lick made of glucose (dextrose) with or without the addition of specific nutrient, prophylactic or therapeutic substances, and including a solution inhibitor for resisting rapid dissolution of the lick, such as chalk or its equivalents, and a nontoxic water-soluble gum or other substance capable of forming a highly viscous solution in water, such as gum acacia, methyl cellulose, methyl ethyl cellulose, sodium carboxy methyl cellulose, sodium alginate, gelatine, sodium pectate and specially treated starches.

The chalk additive is preferably calcium carbonate having a particle size between 1 and 25 microns, and preferably also composed of the calcareous shells of Foraminifera subjected to a method of size reduction to bring the particles down to the size range given without undue destruction of the hollow characteristic of the particles. Such a chalk substance is advantageously incorporated in the block in the proportions of between 2 1/2 and 10% of the total weight of the block.

The procedure employed comprises admixing the additive or additives with a hot glucose liquor, having a high dextrose equivalent and capable of crystallizing on cooling, by agitation in a mixing vessel and, when homogeneity has been attained, discharging the hot liquid into a mold in which it is allowed to crystallize and set to a solid mass. The water-soluble gums are to be added at the rate of 1/2 to 5%.

The effect of the incorporation of chalk in conjunction with a water-soluble gum or substance giving a highly viscous solution in water is that water falling or condensing on the surface of the inhibited block forms initially a thick syrupy solution of glucose, the viscosity of which is further increased by the presence of chalk and resists further dilution and subsequent run-off of the resulting solution. However, the formation of this viscous solution does not reduce the availability of the lick to the animal, as the process of licking involves mechanical abrasion apart from the solvent action of the saliva.

The use of sodium alginate or sodium pectate has a further advantage in that the highly viscous solution formed initially reacts with small quantities of calcium in the lick to form a water-insoluble calcium alginate or pectate gel, which is relatively impervious to water. This soft gel structure is readily removed by an animal's tongue in the act of licking.

In another process R.B. Dawson (U.S. Patent 3,087,820; April 30, 1963; assigned to



Corn Products Co.) describes a lick made only from chalk and glucose.

## Protein Feed Block

As distinguished from pellets or cubes which are relatively small in size, blocks are usually much larger, e.g. 9" x 9" x 12". As a form of animal feed, blocks offer the following advantages:

- (1) Labor saving in that feed blocks will last several days.
- (2) Each animal may get its share and timid or small animals will have an opportunity to eat after more aggressive animals have finished or tired of eating.
- (3) Less bulk and easier to handle, store and feed.
- (4) Savings in feed since no feed blows away or is trampled into the ground.
- (5) Can be fed outdoors without cover except during periods of unusually heavy rainfall and high humidity.

A.J. Gehrt, M.J. Caldwell and W.P. Elmslie (U.S. Patent 2,924,522; February 9, 1960; assigned to Moorman Manufacturing Company) found that alkali treated mucin free vegetable protein materials (e.g. peanut meal, corn gluten meal or soybean flour) could be used very satisfactorily as the basic ingredient of the binder for protein feed blocks, with or without other binder materials such as molasses.

### Example 1 — Preparation of Binder:

| <u>Ingredient</u>               | <u>Percent by Weight</u> |
|---------------------------------|--------------------------|
| Water                           | 78.0                     |
| Sodium hydroxide                | 1.4                      |
| Hydrated lime                   | 0.6                      |
| Vegetable protein (peanut meal) | 20.0                     |
|                                 | <u>100.0</u>             |

The procedure for preparing four hundred pounds of the binder is as follows: place two hundred eighty pounds of water in a tank, add to it eighty pounds of vegetable protein (in the form of peanut meal), and stir these ingredients until complete suspension has been achieved. Add 5.6 pounds of sodium hydroxide dissolved in 20 pounds of water and stir until a uniform consistency has been obtained, then add, with continuous stirring, 2.4 pounds of hydrated lime dissolved in 12 pounds of water. (The order in which these ingredients is added is not important.)

The binder prepared in accordance with Example 1 may be used alone as the sole binder ingredient for a protein feed block, or molasses or a molasses-like material may be used as part of the formulation, primarily for its flavor improving properties.

Example 2 — Preparation of Protein Feed Block:

|  | <u>Percent by Weight<sup>1</sup></u> |
|--|--------------------------------------|
| Feedstuffs                                     | 88 to 94                             |
| Blackstrap molasses                            | 0 to 4                               |
| Vegetable protein binder (as<br>per Example 1) | 6 to 9                               |

<sup>1</sup> As formulated before curing or drying.

The molasses was mixed into the vegetable protein binder portion prepared according to Example 1 and then this mixture was thoroughly blended with the feedstuffs and the resultant blend compressed into protein feed blocks weighing approximately 35 lb. each and measuring 9" x 9" x 12". The feedstuffs used had the following formulation:

|  | <u>Percent by Weight</u> |
|--|--------------------------|
| Protein meals (including soybean,<br>cottonseed, linseed, peanut,<br>wheat germ and corn gluten) | 75 to 85                 |
| Urea (feed grade)  | 3 to 5                   |
| Minerals   | 10 to 20                 |

After the feed blocks have been produced from the foregoing materials as described, they are permitted to dry or cure before shipment or use. These blocks will dry either on standing or, if desired, they may be rapidly dried in a curing room. These blocks showed good resistance to weather and impact.

Nutrient Feed Blocks

A process to provide nutritionally essential nutritive elements in a biochemically acceptable form, fed separately, ad libitum, to the animal so that each animal can obtain its optimum requirement of each element without ingesting unwanted substances has been developed by P.C. Anderson (U.S. Patent 3,198,635; August 3, 1965; assigned to Feed Service Corp.). The nutrient blocks, when licked by the animal, must yield the nutritive element in soluble form so that the animal will taste it and continue ingesting it.

The compositions of this process comprise an intimate mixture of an edible, waxy substance which is solid at ambient temperatures and a member selected from the group consisting of: (1) A saliva-soluble chelate of citric acid and a nutritive element material, and (2) A saliva-soluble mixture consisting essentially of at least one water-soluble citric acid compound and a nutritive element material selected from the group consisting of a metal oxide, a metal hydroxide and a metal carbonate.

One method of preparing nutrient block involves chelating a suitable compound containing the nutritive element in liquid citric acid, allowing the resulting water-soluble chelate complex to harden, and then uniformly dispersing the hardened water-soluble complex in melted edible wax or wax-like material.



Example 1: A nutrient block was prepared from the following materials:

|                              |        |
|------------------------------|--------|
| Manganese carbonate (46% Mn) | 40 g.  |
| Anhydrous citric acid        | 128 g. |
| Food grade stearic acid      | 90 g.  |

The citric acid was melted by heating it to a temperature of 160°C. The powdered manganese carbonate was then rapidly added to the melted acid with vigorous stirring. The temperature and mixing were maintained until the foaming ceased (about two minutes) and the mixture was immediately cooled to solidify the composition. The composition weighed 154 g., indicating a loss of 14 g. The solid citrate-metal chelate composition was then ground to pass through a 20-mesh screen. The waxy food grade stearic acid was heated to 80°C. and the ground chelate was slowly added, with stirring, thereto to form a uniform suspension of the chelate in the waxy material. The resulting mixture was formed into small nutrient blocks as aforesaid.

It is an excellent source of manganese for addition to the diet of an animal, especially when employed in a set and in special feeders, as aforesaid, in accordance with this process. The block was pinkish-tan in color and had a melting point of about 76°C. The composition of this example was odorless to human beings, and possesses all of the desirable properties, such as stability, weather-resistance, palatability and solubility in the saliva of animals. The animal is able to ingest its optimum requirements by licking the block. The block shows no deterioration after extended periods of exposure to climatic conditions.

Another method involves chelating a suitable compound containing a nutritive element in liquid citric acid, allowing the resulting water-soluble chelate complex to harden and then grinding and dispersing, i.e., mixing, it in the waxy substance, in finely divided form, and pressing it into an integral mass.

Example 2: Substantially the same results were obtained when Example 1 was repeated except that the ground chelate was mixed with the waxy substance (food grade stearic acid) and pressed into tablets at the rate of 6 tablets per minute under a pressure of 2,400 lb. per square inch, and then formed into blocks.

A third method for preparing the compositions of this process involves dispersing the aforesaid saliva-soluble mixtures into the waxy substance, which has been melted, thereby forming a suspension, which is then solidified by cooling.

Example 3: A nutrient block was prepared from the following materials:

|                            |        |
|----------------------------|--------|
| Ferrous carbonate (36% Fe) | 92 g.  |
| Ammonium citrate, dibasic  | 135 g. |
| Anhydrous citric acid      | 114 g. |
| Stearic acid               | 113 g. |

The ferrous carbonate, ammonium citrate and citric acid, in finely divided forms, were mixed together and slowly added to melted stearic acid (90°C.) with good stirring, thereby creating a suspension. The final mixture was then poured into paper cartons and allowed to cool.

Thereafter, additional melted stearic acid was poured to a depth of about 2 cm. and allowed to cool to form the base. The block contains 7.3% iron. The block is a dark grey color and has a density of about 1.33. This block has the characteristic mildly acid ferruginous taste, and has excellent resistance to weathering. When heated in an oven, it becomes sticky or tacky at about 75°C. The pH of a 10% aqueous solution of the nonwax fraction is 4.1.

A fourth method in accordance with this process involves pressing together the aforementioned saliva-soluble mixture and waxy substance, which has been finely divided, so as to form an integral mass.

Example 4: A nutrient block was prepared from the following materials:

|                           |        |
|---------------------------|--------|
| Copper hydrate (55% Cu)   | 50 g.  |
| Ammonium citrate, dibasic | 143 g. |
| Citric acid, anhydrous    | 125 g. |
| Stearic acid, food grade  | 136 g. |

The ingredients were all mixed together, ground, and then pressed under 2,600 lb. per square inch pressure in the Stokes punch press, operating at six tablets per minute. Three tablets were dipped in melted stearic acid, as was a disc of plywood of the aforementioned dimensions. The three tablets were stacked over the coated plywood disc and allowed to cool, whereupon a block was formed by the aggregation of tablets and plywood. The block contains 6.17% copper, has the characteristic acid copper taste, and is bluish green color. Its density is about 1.10. When heated it becomes sticky or tacky at about 75°C. Similarly nutrient feed blocks containing cobalt, nickel, zinc, calcium and magnesium can be prepared.

A sweetening agent can be added to the nutrient block to overcome the acid taste cause by the low pH of the composition, (sucrose or saccharin). In use, the compositions are formed into blocks and presented to the animals in suitable feeding devices so designed as to permit the animal to lick the surface but to prevent it from biting off chunks or pieces.

## DETOXIFYING PLANT MATERIAL

### Removing Toxins from *Agave Lecheguilla*

The plant *Agave lecheguilla* grows profusely in the semiarid regions of certain parts of Texas and New Mexico. Following the ingestion of this plant material, sheep and cattle develop the signs and symptoms of the disease called bighead. The economic loss of poisoned animals and unutilizable waste land represents considerable hardship to the inhabitants of those regions.

M. Rubin (U.S. Patent 3,069,269; December 18, 1962; assigned to Advance Growth Capital Corporation) found that by suitable extraction procedures the toxic ingredients can be removed from the *Agave lecheguilla*. The residual plant material remaining after extraction of the toxins has all the nutritive values of the plant. Concentration of the extracts obtained from *Agave lecheguilla* by the procedure which follows provides a residue with unusual detergency properties. The extract product is largely a saponin and as a by-product could be converted to steroid pharmaceutical products.



Example: 300 lb. of meal obtained by mechanical decortication of the Agave lecheguilla plant were extracted by stirring at 40° to 50°C. with 300 gal. of 95% ethanol. The mixture was stirred for 1 1/2 hours and then the solvent removed by decantation and filtration. The extraction procedure was repeated twice more with equal quantities of 80% ethyl alcohol. Following the last extraction, decantation and filtration, the residual meal was dried in a rotary drier at 75°C. with provision for recovery of the extracting solvent. The resulting dry meal so obtained constitutes a well blended particulate mixture of excellent suitability for grinding to feed consistency in a Wiley mill. Analysis of material prepared in this manner provided the following proximate analytical results:

|                                 |        |
|---------------------------------|--------|
| Dry matter                      | 93.1 % |
| Protein                         | 7.33%  |
| Ash                             | 19.8 % |
| Fat                             | 0.54%  |
| Crude fiber                     | 22.0 % |
| Readily digestible carbohydrate | 50.4 % |
| Total digestible nutrients      | 55.0 % |

It is evident from these results that the product has the feed equivalent of cured dried hay.

### Treating Oleaginous Substances to Reduce Growth Inhibitors

L.G. Wenger and L. Wenger (U.S. Patent 3,385,709; May 28, 1968; assigned to Wenger Manufacturing, Inc.) have developed a process for efficiently treating oleaginous agricultural products such as seeds or beans having growth inhibition material therein, to effectively destroy the inhibitors so that the products may be fed to various types of animals.

Briefly, the process contemplates the grinding or flaking of whole oleaginous seeds or pulses by some appropriate means and thereafter advancement of the ground oleaginous agriculture products to be treated, continuously along a predetermined path of travel through treatment equipment in a manner to efficiently and quickly raise the temperature of the products to a level sufficient to destroy or reduce the effects of growth inhibition material contained in the products but without adversely affecting the nutritive value of the agricultural products.

In order to rapidly raise the temperature of the products to the required level, the oleaginous substance is passed through a first preconditioning zone which is steam jacketed and has means for introducing a quantity of steam (5 to 10%) into the products during agitation and advancement thereof so that the products are raised to a temperature of approximately 180° to 210°F. in a very short interval of time (1 1/2 to 3 minutes). Next, the heated products are passed through a second high speed mixing and conditioning zone where the temperature thereof is maintained at approximately 200° to 215°F. and with relatively rapid agitation and tumbling of the material being effected in the second conditioning zone to assure heating of all of the product to a uniform temperature.

Finally, the product is forced through a third zone comprising an extrusion press fitted with a conical member to increase the pressure on the product sufficiently to raise the temperature thereof to at least approximately 250° to 300°F. in 7 to 30 seconds in order to destroy or reduce the effects of the growth inhibiting material contained in the agricultural product and



with the pressure on the substance being released as the same emerges from the conical member. The final material, which emerges from the machine in the form of one or more extruded ribbons, may be cut into suitable pellet size for direct feeding to animals or may be reduced in particle size thereafter to meal or granular form and mixed thereafter with other feeds if desired.

### Detoxified Meal from *Crambé Abyssinica* Seed

A method of processing the seeds of *Crambé abyssinica* to remove known metabolic toxicants and provide an excellent source of palatable protein having use as a basic feed for livestock has been developed by G.C. Mustakas and L.D. Kirk (U.S. Patent 3,173,792; March 16, 1965; assigned to the U.S. Secretary of Agriculture).

Example: *Crambé abyssinica* seed (5,200 g.) was cracked between corrugated rolls, dehulled by aspiration, tempered to 9% moisture, and rolled between smooth rolls to produce flakes having an average thickness of 0.002 to 0.005 inch. About 4,000 g. of the flakes were charged to a converter-cooker equipped with an agitator to provide thorough mixing. While stirring the charge at room temperature, moisture was added to provide a total moisture content of 30%. The moist flakes were heated to 50°C. (130°F.) and held at this temperature for 15 minutes to promote enzymatic hydrolysis of the thioglycosides to thiooxazolidone and isothiocyanates.

At the end of this period, open steam was added to the charge through a perforated coil sparger and at the same time heat was applied indirectly through a jacket containing steam at 30 psig. The charge was steamed for 30 minutes and then dried to approximately 19% moisture using indirect heat only. After removing the charge and cooling with air, the crisped material was passed through smooth rolls twice at 0.001 inch clearance and was then subjected to a miscella extraction with hexane for removal of the triglycerides.

The slurry was then filtered and the cake was washed successively with hexane washes containing 5, 1, and 0% oil content, each weighing 5,200 g. After addition of the final wash the cake was drained under vacuum for 10 seconds and air dried to produce approximately 2,200 g. of meal. Whereas the untreated raw meal had analyzed 0.058% of isothiocyanate (calculated as allyl isothiocyanate) and 0.897 of 5-vinyl-2-thiooxazolidone, the detoxified meal analyzed 0.005% and 0.000 respectively.

### Microbiological Decontamination of Aflatoxin

The aflatoxins constitute a group of apparently carcinogenic mycotoxins formed by the growth of certain *Aspergilli* on exposed moist cereal grains, peanuts, cottonseed, and related agricultural materials commonly used as foods or in animal feeds. A. Ciegler and E.B. Lillehoj (U.S. Patent 3,428,458; February 18, 1969; assigned to the U.S. Secretary of Agriculture) have a process for completely eliminating the aflatoxins from contaminated foods and animal feed materials comprising exposing the contaminated material for some hours to contact with a sufficient number of viable cells of the nonpathogenic and completely harmless bacterial organism *Flavobacterium aurantiacum* NRRL B-184.

Since *Flavobacterium aurantiacum* NRRL B-184 is nonpathogenic, the intact cells per se are



unobjectionable in materials that may be employed in the feeding of livestock, poultry, etc. On the other hand, where the cells might possibly be objected to on purely philosophical grounds, as in foods for human consumption, they are readily destroyed by pasteurization or sonication.

Test Procedure: Flavobacterium aurantiacum NRRL B-184 produces nonfluorescing zones of inhibition on specially formulated agar plates, such as a modified Czapek-Dox medium having the following composition per 1,000 ml. that per se exhibited no fluorescence prior to the addition (to the hot solution) of 1 ml. of chloroform containing 0.25 mg. aflatoxin B<sub>1</sub>.

|                                     |         |
|-------------------------------------|---------|
| Sucrose                             | 30.0 g. |
| NaNO <sub>3</sub>                   | 3.0 g.  |
| K <sub>2</sub> HPO <sub>4</sub>     | 1.0 g.  |
| MgSO <sub>4</sub>                   | 0.5 g.  |
| KCl                                 | 0.5 g.  |
| FeSO <sub>4</sub>                   | 0.01 g. |
| "Bacto-Yeast Extract"               | 0.05 g. |
| Agar                                | 20.0 g. |
| Distilled water q.s. ad 1,000.0 ml. |         |

TLC quantitation [method of Delongh et al, Biochem. Biophys. Acta 65:548 (1962)] of the substantial detoxification by Flavobacterium aurantiacum of a liquid system that was expected in view of the Petri dish evidences of zonal inhibition was obtained upon incubating  $2 \times 10^{13}$  resting viable cells of Flavobacterium aurantiacum NRRL B-184 for about 44 hours at 200 shakes per minute and at 28°C. in 300 ml. Erlenmeyer flasks containing 50 ml. of a modification of Haynes' et al TGY broth [Appl. Microbiol. 3:361 (1955)], the modified broth having the following composition per 1,000 ml.:

|                                 |         |
|---------------------------------|---------|
| "Bacto-tryptone"                | 2.5 g.  |
| "Bacto-Yeast Extract"           | 2.5 g.  |
| Glucose                         | 10.0 g. |
| K <sub>2</sub> HPO <sub>4</sub> | 1.0 g.  |
| Tapwater q.s. ad 1,000.0 ml.    |         |

to which flasks of inoculated medium also had been added 1 ml. of chloroform containing 0.5 mg. or 1.0 mg. of either aflatoxin B<sub>1</sub> or G<sub>1</sub>.

Example 1: This example was designed to simulate the natural occurrence of aflatoxin contamination in an animal feed material and the effective decontamination thereof. 50 g. of undiseased cracked whole soybeans in a 500 ml. Erlenmeyer flask were moistened with 10 ml. of water, and the mouth of the flask plugged with cotton. The flask was autoclaved at 121°C. for 30 minutes and when cool the sterilized beans were inoculated with a heavy suspension of spores of Aspergillus flavus. After incubation at 28°C. for 7 days, the flask was autoclaved to kill the mold. The contaminated soybeans were vacuum dried at 60°C. for 16 hours, and ground to a meal in a Wiley mill. By TLC analysis of a chloroform extract of a 2.5 g. sample, it was found that each gram of the soybeans contained 6 µg. of aflatoxin B<sub>1</sub> and the same amount of aflatoxin G<sub>1</sub>. A 2.5 g. sample of the contaminated soybeans was mixed with 22.5 g. of uncontaminated soybeans, and to the mixture was added 5 ml. of 0.9 NaCl

solution containing  $1 \times 10^{13}$  viable cells of Flavobacterium aurantiacum. After 16 hours of incubation at 28°C. the incubated mixture was extracted with 400 ml. chloroform, the extract filtered through glass wool, the filtrate evaporated to 10 ml., and the concentrate assayed for aflatoxin by the previously described TLC method. No residual aflatoxin was detected.

Example 2: To biologically confirm the removal or destruction of aflatoxins by Flavobacterium aurantiacum,  $8 \times 10^{12}$  viable cells of Flavobacterium aurantiacum were added to 80 ml. of buffered very faintly acidic solution containing 600 µg. of aflatoxin B<sub>1</sub>, and the mixture was incubated at 28°C. for 12 hours. A parallel handling with aflatoxin G<sub>1</sub> was also performed. After chemically verifying the total inactivations of the aflatoxins, the inactivated samples along with samples of the aflatoxins themselves and a separate sample of the cells were sent to the Wisconsin Alumni Research Foundation which on a fee basis conducts the duckling bioassays based on the absence or the development of bile duct hyperplasia upon autopsying 8 day ducklings that were fed the suspected material 1 week prior. The impartial data reported is presented below.

| Material fed to 1-day ducklings                           | Total aflatoxin content in µg. per duckling | Bile duck hyperplasia on autopsy on 8th day |
|---|---|---|
| Aflatoxin B <sub>1</sub> .....                            | 8.0   | +   |
| Aflatoxin plus <i>F. aurant.</i> cells.....               | ( <sup>1</sup> )                            | —   |
| Aflatoxin G <sub>1</sub> .....                            | 7.0   | +   |
| Aflatoxin G <sub>1</sub> plus <i>F. aurant.</i> cells.... | ( <sup>2</sup> )                            | —   |
| Aq. dispersion of <i>F. aurant.</i> cells....             | 0   | ( <sup>3</sup> )                            |

<sup>1</sup> (None) if not deactivated, the 7.5 ml. would have contained a total of 52.5 µg. aflatoxin B<sub>1</sub>.

<sup>2</sup> (None) if not deactivated, the 7.5 ml. would have contained a total of 30.0 µg. of aflatoxin G<sub>1</sub>.

<sup>3</sup> Nontoxic control.



# FATS AND OILS

## REFINING OF VEGETABLE GLYCERIDE OILS

Crude glyceride oils are extracted from seeds, nuts or beans by pressing or the action of solvents or by both. Fatty oils are ordinarily refined by adding to the oil an aqueous alkaline solution, followed by conditioning or other treatment, and then by separating from the oil the soapstock formed. The purpose of such refining is to neutralize and remove the free fatty acids present, to remove whatever gums may be present, to remove impurities and/or to improve oil color. The losses that occur during the alkaline refining of fatty oils result from: (1) The entrainment of neutral fatty oil in the soapstock formed; (2) The absorption of neutral fatty oil in the gums which separate along with the soapstock; (3) The saponification of neutral fatty oil into soapstock; (4) The emulsification of neutral fatty oil in the soapstock phase.

### Use of Sodium Carbonate

A.U. Ayres and F.H. Smith (U.S. Patent 2,838,553; June 10, 1958; assigned to Sharples Corporation) describes a process for refining fatty oils.

Example 1: 12 tank cars of a highly colored cottonseed oil having an average free fatty acid content of 1.6% by weight was refined in a continuous vented stream under temperature conditions between 200°F. and 210°F. with an aqueous sodium carbonate solution of 26° Bé. The amount of sodium carbonate employed was between 2 and 3 times the theoretical stoichiometric amount, the average being 2.1 times. After separation of the soapstock from the oil by centrifuging, the oil upon analysis showed an average free fatty acid content of 0.06%. The separated oil was re-refined in a continuous stream under temperature conditions between 155°F. and 160°F. with 3% by weight based on the oil of an aqueous caustic soda solution of 20° Bé. After separation of the reagent from the oil by centrifuging, the oil upon analysis showed an average free fatty acid content of 0.02%. The oil was then subjected to conventional water washing and vacuum drying.

The overall loss was 6.1% by weight based on the original oil, and the final refined oil had an average bleach color of 20 yellow, and 2.4 red, which is an outstanding result since it exemplifies the refining of highly colored cottonseed oil into what is known in the trade as a prime oil, i.e., an oil having a bleach color not greater than 2.5 red, which is an extremely difficult task as exemplified by the color obtained through laboratory refining as noted below. The average laboratory "refining loss" as determined analytically, employing the applicable official method of the American Oil Chemists Society, was 8.1% by weight,

and the average bleach color so determined was 20 yellow and 2.7 red. The outstanding results obtained are pointed up by the fact that it is extremely difficult in the case of a highly colored cottonseed oil to reduce the red bleach color below that obtained by the above official method.

Any other fatty oil may be substituted with comparable results. The centrifugal forces used in the separations effected were between approximately 10,000 and 15,000 times gravity. Also full bowl separation may be substituted for the open bowl separation, although the open bowl separation with the atmospheric venting employed is preferred. Likewise, separation by gravity may be substituted, but with less efficient separation.

### Removal of CO<sub>2</sub> Before Oil-Soapstock Separation

A process for the refining of glyceride oils, by use of soda ash in such manner that carbon dioxide is evolved and removed before the oil-soapstock mixture is separated, has been developed by B.H. Thurman (U.S. Patent 2,876,242; March 3, 1959; assigned to Benjamin Clayton). An important advantage of the process is that it can produce a soapstock excellently suited as an additive to animal feeds. For example, the low-excess soda ash soapstock resulting from refining a crude soya oil may be used instead of lecithin for feeds.

Figure 3.1 is a schematic pipe-line diagram of an apparatus suitable for carrying out a continuous process. This exemplified apparatus includes, generally described, a proportioning heating means 10, a degasifying means 12, a separating means 14 and a re-refining means 16. The oil to be refined may be withdrawn from a tank 18 by a proportioning pump 19 which delivers a stream thereof through a heater 20 to a mixer 21. Similarly, a solution of soda ash may be withdrawn from a tank 22 by a proportioning pump 23 which forces the stream through a heater 24 and through a pipe 25 which may discharge directly into the mixer 21 or, as shown, into a pipe junction 26 to preliminarily mix with the oil just ahead of the mixer. The oil-soapstock mixture issuing from the mixer 21 is advanced, under the pressure imposed by the pumps 19 and 23, through a heater 32 from whence it is delivered to the degasifying means 12 through a pipe 33. The heaters 20, 24 and 32, as well as similarly-shown heaters to be later mentioned, are each illustrated as including a coil 34 in a housing 35 through which any desired heating medium is circulated by means of pipe connections 36 and 37.

The degasifying means 12 includes, collectively or singly, gas separators 40 and 41 sequentially connected between the proportioning-heating means 10 and the separating means 14. These gas separators collectively or singly provide a first zone in which carbon dioxide is separated from the oil-soapstock mixture and removed from the system. The separating means 14 provides a second zone in which the soapstock is separated from the oil. The oil-soapstock mixture flowing through the pipe 33 under the control of a valve 43 enters a container 44 of the gas separator 40 at a level several inches beneath the surface of a body of oil-soapstock mixture 45 therein. The discharge is typically at a position about  $1/4$  to  $1/2$  the height of the container 44 measured from its lower wall. A stream of the oil-soapstock mixture from the body 45 is withdrawn, usually continuously, from the lower end of the container 44 through a pipe 46. The upper end of the container 44 is filled with a body of carbon dioxide 45a which blankets the surface of the body 45. Carbon dioxide is continuously evolved and separated from the body 45 of constantly-renewed oil-soapstock mixture in the container 44.

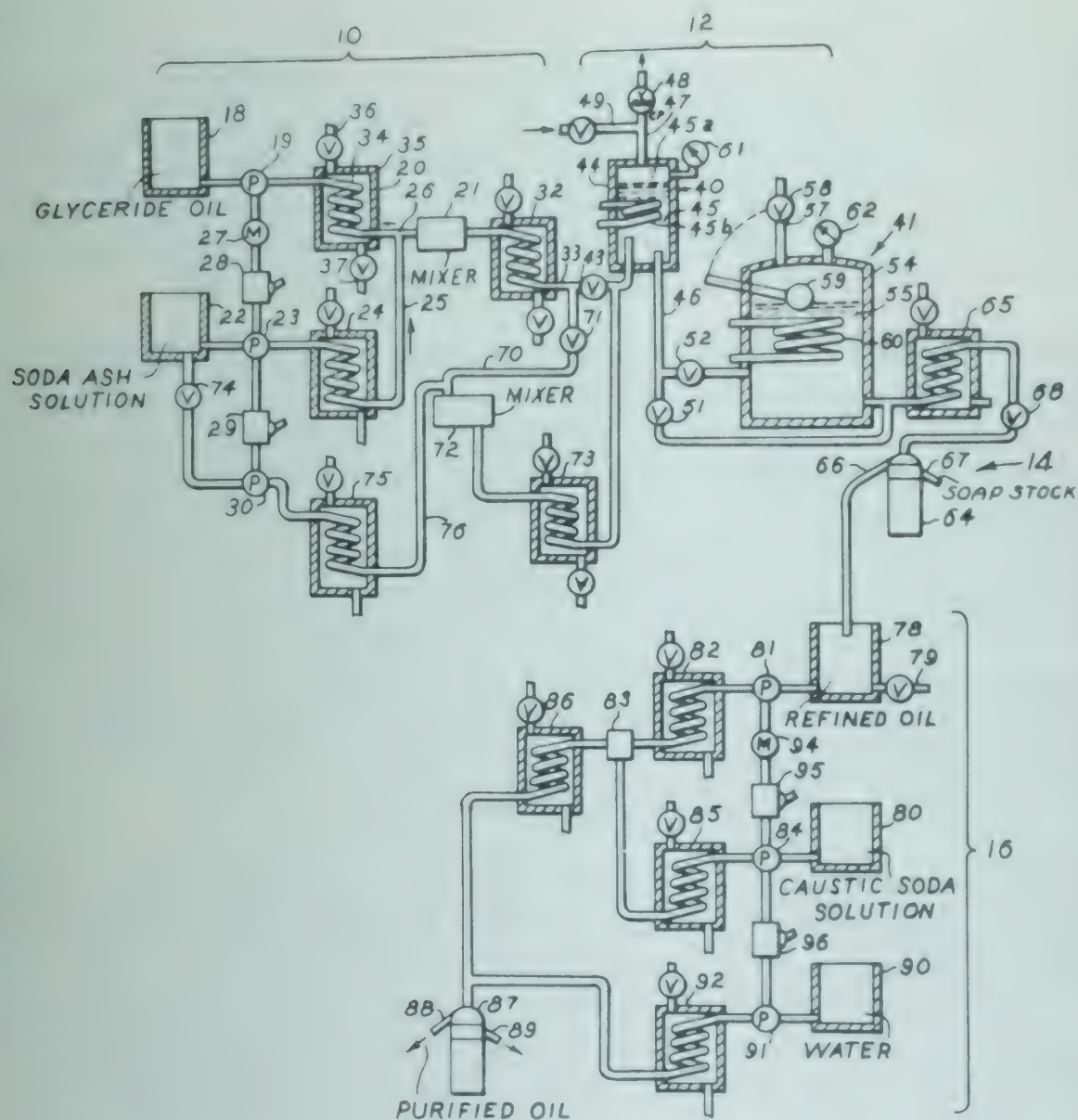


If desired, this body may be heated by a heater 45b. Means is provided for maintaining a substantially constant pressure in the container 44, preferably a pressure which is above atmospheric and above the pressure in the separating means 14 by a predetermined degree. In some instances, this can be accomplished by discharging a continuous stream of the carbon dioxide from the closed container 44 through a restriction such as provided by a pipe 47, permitting this gas to discharge into the atmosphere. It is preferable, however, to dispose a pressure-regulated valve 48 in the pipe 47, this valve controlling the flow of carbon dioxide to maintain the pressure in the container 44 substantially constant. A valve pipe 49 may communicate with the pipe 47 to supply compressed air, carbon dioxide or other gas to the upper interior of the container 44 to build up an initial pressure therein during starting of the apparatus and before the body of carbon dioxide 45a is produced.

By opening a valve 51 and closing a valve 52, the degasified oil-soapstock mixture from the gas separator 40 can be delivered directly to the separating means 14 to by-pass the gas separator 41. However, it is often desirable to close the valve 51 and open the valve 52 to flow this mixture through the gas separator 41 wherein additional carbon dioxide may be liberated and wherein a constant head can be imposed on the mixture to feed it at a uniform rate to the separating means 14. The gas separator 41 includes a closed container 54 in which a body 55 of the oil-soapstock mixture collects. Any carbon dioxide evolved and separated from this body is removed through a pipe 57 under the control of a valve 58. This valve is operatively connected to a float 59 to maintain substantially constant in vertical position the level of the body 55. A heater 60 may be installed in the container 54 to heat or maintain the temperature of the body of mixture therein. The pressures in the containers 44 and 54 are indicated respectively by gauges 61 and 62.

The float-operated valve 58 serves also the desirable function of preventing the surface of the oil-soapstock mixture from rising to the intake of the pipe 57, thus acting as a means for maintaining a body of separated carbon dioxide in the container 54 at all times. It thus prevents filling and overflowing of the chamber and coating of the valve parts with the oil-soapstock mixture. A similar float-operated valve is desirably used with the container 44 if only the gas separator 40 is employed ahead of the separating means 14, such a valve being employed as a substitute for or as a supplement to the pressure-regulated valve 48, the latter being preferred. The separating means 14 is preferably one or more centrifuges. Conventional centrifuges can be used which operate at substantially atmospheric pressure; centrifuges can be of the closed or hermetic type, operating at superatmospheric pressures and with the effluent streams of substantially neutral oil and soapstock maintained under pressure after they have discharged from the centrifuge.

The degasifying of the process aids in the soda ash refining using such centrifuges as it irons out pulsations and avoids surging therein, particularly at temperatures of 160°F. or higher at the entrance to the centrifugal separator. A conventional centrifuge 64 operating at atmospheric pressure and receiving the degasified soapstock-oil mixture through a heat exchanger 65 which may be optionally used or bypassed. This centrifuge separates the mixture into a stream of substantially neutralized oil, discharging through a pipe 66, and a stream of soapstock, discharging through a pipe 67. A valve 68 is preferably disposed in the line ahead of the centrifuge 64 and with the valves 43 and 51 or 52 can be adjusted to regulate the pressures in the various chambers and lines.

FIGURE 3.1: REMOVAL OF CO<sub>2</sub> BEFORE OIL-SOAPSTOCK SEPARATION

Source: B.H. Thurman; U.S. Patent 2,876,242; March 3, 1959

The process operates best at superatmospheric temperatures whether the soda ash is added in one or two portions. The temperatures at the time of separation in the centrifuge 64 are desirably in the neighborhood of 190° to 220°F., usually about 190° to 210°F. The heater 65 may be used to supply some of this heat or merely to maintain the temperature of the oil-soapstock mixture fed therethrough from the gas separators 40 or 41. The temperature in these separators is substantially the same as the temperature of the oil-soapstock mixture fed thereto, this temperature ordinarily being about 160° to 220°F., albeit not above the boiling point of water at the pressures existing in the gas separators 40 or 41, as it is not desired to dehydrate the oil-soapstock mixture by removing any substantial amount of water therefrom. This amount of heat can be supplied almost entirely by the heater 32, sometimes supplemented



by the heater 73 or by the heating coils 45b or 60.

The pressure in the gas separator 40 is preferably superatmospheric, the discharge of carbon dioxide being controlled or restricted to create sufficient pressure to advance the oil-soap-stock mixture to the centrifuge 64. Pressures in the container 44 are preferably 5 to 20 psi, usually about 5 to 10 psi although they may be as high as 50 psi or higher under some circumstances.

### Oil Extraction with Aqueous Ammonia

B. Clayton (U.S. Patent 2,939,790; June 7, 1960) has an improved process for refining glyceride oils in which a small amount of an aqueous solution of ammonia is employed as the refining reagent.

Example: A solvent free solvent extracted crude soya bean oil containing 0.85% free fatty acids by weight and having a Wesson loss of 2.2%, i.e. having a gum content of 1.35% by weight, was refined employing 1.08% water and 0.18% ammonia, both by weight based on the oil, i.e. 1.26% of a 14% ammonia solution. The percentage of solution employed is based on the weight of the oil and the percentage of ammonia in the solution is based on the weight of the solution. The amount of ammonia can be stated as 18% of the free fatty acids or 0.15% of the oil plus 2.25% of the gums or 0.03% of the oil or a total of 0.18% of the oil. The amount of water was 49% of the total weight of the free fatty acids and gums. The crude oil was heated in a storage tank to 140°F. and was delivered through a pump and meter to the mixer. The ammonia solution was also delivered through a pump and meter to the mixer and initial mixing was merely by flowing the streams together. The initial mixture was passed through a vertical mixer provided with a plurality of spaced paddle blades extending horizontally from a vertical shaft and rotated thereby so as to subject the oil to relatively mild mixing, the time in the mixer being approximately 6 minutes.

The temperature of the resulting mixture was a few degrees below 140°F. and was sent through a heat exchange in indirect heat exchange with a heating medium to raise its temperature to 140°F. The heated mixture was delivered into a conventional open type centrifuge and continuously centrifugally separated into a neutral oil and ammonia foots or soap-stock. A centrifuge of the sealed or vapor-tight type might be advantageously used at this point. The refining loss in the ammonia refining step was approximately 2.38% i.e. not much greater than the Wesson loss. The separated oil contained 0.11% free fatty acids and 0.07% gums. The foots or soapstock had a moisture and volatile content of 21.3% based on the total weight of the wet ammoniated foots and contained 14.0% free glyceride oil and 12.6% free fatty acids on a dried and deammoniated basis, the remaining 73.4% being mostly phosphatides with small amounts of other materials including vitamin B complex.

The wet foots, when added directly to a seed meal such as the soya bean meal resulting from the recovery of the original soya bean oil and the resultant mixture dried, imparts increased nutritional values and improves the appearance and physical properties of the meal. Crude cottonseed oil could similiarly be used.



Using Sequentially Caustic and Soda Ash

In this process, B. Clayton (U.S. Patent 2,991,178; July 4, 1961) had found that refining by the joint use of caustic and soda ash can produce unexpected benefits if the caustic is employed in carefully limited amount. If the two alkalis are employed sequentially, the resulting soapstock makes an excellent feed additive. The gums therein are intact in the sense of being substantially free of alkali degradation. Best results have been obtained by mixing with the oil an amount of caustic equal to about 50% to 85% of that amount that would be necessary to neutralize the free fatty acids of the oil. The remaining or residual fatty acids are then neutralized by the later-added soda ash solution, with many attendant advantages. Best results follow from the use of an amount of soda ash calculated on the following basis:

$$\text{Percent dry soda ash} = 0.075 - 0.15 \times \text{Wesson Loss}^* \text{ of crude oil}$$

(\*The Wesson Loss is a well known property of glyceride oils, being expressed in a percentage figure and being determined by the method described on pages 400-402 of "Vegetable Fats and Oils" by G.S. Jamieson, published in 1932 by The Chemical Catalog Company, New York, N.Y.)

Example 1: An example in which the formula is applied is as follows: A crude soya oil containing 0.5% free fatty acids and 1.5% gums and having a Wesson Loss of 2.0% can be re-refined in accordance with the invention by adding 0.15% of a 30° Bé. (24%) solution of caustic soda, representing 50% of the amount required to neutralize the free fatty acids. The amount of soda ash to add, using a multiplication factor of 0.1 in the above equation, is 0.20% dry soda ash. This corresponds to 1.33% of a 20° Bé. soda ash solution. The soapstock from this refining will contain about 30.8% moisture and 20.0% free oil, dry basis.

Example 2: As an example of a process employing caustic soda solution in amount about 70% of that required to neutralize the free fatty acids, a crude cottonseed oil having 2.7% free fatty acids, a Cup Loss (by the Official Method, American Oil Chemists' Society) of 9.7% and a Wesson or Absolute Loss of 4.5% was refined with 1.12% of 30° Bé. caustic soda solution proportioned in flow into the crude oil at a temperature of 90°F. The solution was very rapidly and intimately mixed, using a pipe-line mixer with its agitator rotating at a speed of 3600 rpm. After a residence time in flow of 20-30 seconds in the larger pipe section, 2.6% of 22° Bé. soda ash solution (17.5% Na<sub>2</sub>CO<sub>3</sub>) was proportioned into the mixture, the resulting mixture being quickly mixed in a similar pipe-line mixer and then heated to 190° to 200°F. in the heat exchanger. The resulting centrifuged oil was re-refined with 1.25% of 30° Bé. caustic soda, water-washed and vacuum-dried. The refining loss was 5.63%, which loss was 42.0% lower than the official Cup Loss. The entire soapstock separated in the centrifuge is desirably returned to the meal ahead of the desolventizer or drier. The amount of soapstock in the meal was about 2.75% by weight.

Low-Excess Soda Ash Soapstock

B.H. Thurman (U.S. Patent 2,968,559; January 17, 1961; assigned to Refining Inc.) has found that in refining crude glyceride oils if the amount of soda ash is in low excess, e.g., no more than about three times the amount required to react with the free fatty acids, there is substantially no alkali degradation of the phosphatides. These soapstocks are suitable feed



additives. In general, the addition of low-excess soda ash soapstocks to meals to form a feed product has many advantages. Minor amounts of such soapstocks will decrease dusting and markedly better the pelletizing properties of meal while also bettering the color, increasing the nutritive qualities, making the food product more palatable, and stimulating the appetite. The feed products of the process when ingested by cattle disperse quickly in the stomach or paunch, the soapstock serving to keep the meal or other ingredients suspended so that digestion proceeds uniformly. The condition known as "bloat", in which the feed products sink to the bottom of the stomach and gas accumulates on top thereby choking the animal is entirely avoided.

Example 1: The process will be first exemplified as employing soda ash mixed with the oil in amount not substantially in excess of three times the amount necessary to neutralize the free fatty acids of the oil and preferably in amount ranging from a fraction of that required to neutralize up to about twice that required to neutralize. In a typical operation, soya beans were solvent extracted to produce a crude soya oil containing 1.41% free fatty acids and very high in phosphatides. The resulting crude oil was partially refined by preheating it in flow to 180°F. and mixing therewith an amount of soda ash substantially equal to 1.5 times the amount required to neutralize the free fatty acids, the soda ash being added as a 20° Bé. solution. The mixture was cooled to about 115°F. and separated in a suitable centrifuge. The separated low excess soda ash soapstock contained about 25.0% free oil, dry basis, and about 20.0% water, the remainder being gums, soaps resulting from the reaction of the soda ash and the free fatty acids, and a small amount of free alkali. The soapstock assayed 1.92% phosphorus, dry basis. Three ounces of such low excess soda ash soapstock added daily to a control meal showed average weight gains in heifers of 87.5 lbs., as against a gain of 65 lbs. on heifers feeding on the control ration, in a test of four weeks duration. When cottonseed oil soapstocks or meals are used levels of free gossypol which is toxic to animals must be reduced.

Example 2: To 100 parts of a solvent-extracted cottonseed meal containing 0.41% free gossypol, dry basis, was mixed 3.0 parts, dry basis, of a low excess soda ash cottonseed soapstock containing 1.44% free gossypol, dry basis. The soapstock was in this instance mixed with 15 parts of water before being added to the already desolventized meal. The mixture was placed in a jacketed closed-type mixer. After mixing for a period of about 15 minutes at room temperature, the temperature in the jacket was raised rapidly to 107° to 110°C., the mixing being continued for 30 minutes at this temperature. Thereafter, the mixing vessel was opened and the excess moisture evaporated from the mixture during a 15 minute additional mixing time. The resulting feed product contained 0.026% free gossypol, corresponding to 0.028% dry basis.

### SOAPSTOCKS IN FEED PRODUCTS

"Soapstock" may be defined as the residue obtained in the alkaline refining of vegetable oil, and this contains a saponified fatty acid fraction, a phosphatide and sterol fraction together with some of the pigments of the oil. It is a high energy source, containing fatty acids. The residue material is emulsified and alkaline in character, has surfactant properties, and has a very high content of xanthophyll.

Detoxified Cottonseed Oil Soapstock

In a process developed by F.C. Pack and L.S. Goldblatt (U.S. Patent 2,746,864; May 22, 1956; assigned to U.S. Secretary of Agriculture) cottonseed oil foots or "soapstock" are converted to non-toxic cottonseed oil foots containing less than about 0.02% free gossypol by heating cottonseed oil foots in a closed vessel to a temperature of from about 210°C. to 220°C. for from about 1 to 3 minutes. Samples of cottonseed oil foots obtained by a conventional soda ash refining of crude cottonseed oil were heated in closed vessels for the indicated times at the indicated temperatures. The effects of the treatment upon the gossypol content of the foots is summarized in the following table.

|                          | Percent Gossypol |       |
|--------------------------|------------------|-------|
|                          | Free             | Total |
| Control.....             | 3.76             | 4.20  |
| 180° C. (4 minutes)..... | 0.54             | 0.67  |
| 190° C. (4 minutes)..... | 0.15             | 0.16  |
| 200° C. (1 minutes)..... | 0                | 0     |
| 210° C. (2 minutes)..... | 0                | 0     |
| 220° C. (1 minute).....  | 0.30             | 0.44  |
| 220° C. (2 minutes)..... | 0                | 0     |
| 240° C. (1 minute).....  | 0.15             | 0.32  |

In the production of the oilseed meal composition, any of the oilseed meals adapted for use as animal feedstuffs can be used. The use of detoxified cottonseed oil foots in proportions of from about 1 to 10 parts by weight of foots per 100 parts of meal, is preferred.

Soapstock Meal Blend

N.H. Witte and E. Sipos (U.S. Patent 3,033,683; May 8, 1962; assigned to Central Soya Company, Inc.) have developed a process for incorporating soybean soapstock into an oilseed meal. The soybean flakes must first be steam and heat treated to inactivate enzymes present which would destroy xanthophyll in soapstock. Examples of the processes may be set out in a more specific form as follows.

Example 1: A sample of steam treated and deodorized extracted soybean flakes was treated with 20% soybean soapstock (moisture content of 47%) and with live steam for 15 minutes in a laboratory toaster provided with stirring paddles. The live steam not only served to raise the temperature of the mixture, but also the condensation of the steam was effective in driving the soapstock into the interior of the meal particles by virtue of lowering its viscosity. The subsequent drying was found to seal in the soapstock within the particles and to give a product having good flow properties. The meal protein also reacted with the soapstock so that most of the fatty acids were made available for fat extraction with petroleum ether or diethylether. The finished product analyzed 7.1% fat and 61 mcg./gm. of xanthophyll.

Example 2: The process of Example 1 was followed except that 1% isopropyl alcohol was added to the soapstock prior to mixing it with the meal in order to reduce the viscosity of the soapstock for better spreading action. The viscosity of the soapstock was reduced from 65,000 centipoises at 30°C. to 21,000 centipoises at 30°C. The sample was mixed with



much greater ease than in the case of Example 1, and in every other respect it was similar to that obtained in Example 1. In addition to isopropyl alcohol, ethyl, propyl and butyl alcohols can also be used successfully.

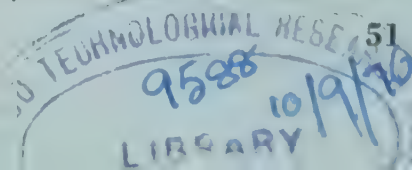
Example 3: A sample of steam treated and deodorized extracted soybean flakes was treated with 30% partially acidified soybean soapstock (moisture content of 40 to 60%) and with live steam for 15 minutes in a laboratory toaster. Acidification was achieved in a mixer by the addition of 5% concentrated sulfuric acid to a previously heated and well agitated soapstock. The resulting acidified material, having a pH of 5.0 to 6.5, is easily spread out on the surface of meal particles because of its fluid state, in contrast to the gelatinous consistency of raw soapstock and the hard, gummy characteristics of a normal soapstock. This partially acidified soapstock is superior to a completely acidified soapstock because it gives greater stability to the xanthophyll pigments present in this material, which are sensitive to very acidic pH's. While the pH range 5.0 to 6.5 affords good xanthophyll stability, it also causes extensive hydrolysis of the soap with the subsequent liberation of fatty acids that are very low in viscosity, and are easily soaked into the meal particles.

When toasting and drying drives off the excessive liquids, the fat is sealed within the meal particles to produce a dry exterior texture and excellent flow properties similar to that claimed for animal and vegetable fats. Furthermore, such a product is an excellent pigmentation source for broilers because it can contain 80 to 100 mcg./g. of xanthophyll. The xanthophyll in this "soapstock meal" also has good storage stability due to the high tocopherol content of soybean soapstock and due to the close to neutral pH of the finished product. While soapstock cannot be extracted as fat by the conventional analytical methods used in the trade, partially acidified soapstock could be recovered up to 80% to 90% on dry basis from the meal which is the equivalent of the fatty acid content of soapstock. The addition of 30% partially acidified soapstock to soybean oil flakes resulted in a fat content of 12 to 14% in the finished product.

### Lecithin Products from Soapstocks

B.H. Thurman (U.S. Patent 2,970,910; February 7, 1961; assigned to Refining Unincorporated) has a process to refine crude glyceride oils in such way as to produce a valuable lecithin product. The soapstocks with which this process is concerned are those resulting from the refining of crude glyceride oils by non-saponifying, non-volatile alkalis so that the soapstocks still contain the indigenous phosphatides substantially unmodified. Such soapstock is acidulated with only sufficient mineral acid largely to convert any excess soda ash to sodium sulfate, the soaps to fatty acids and sodium sulfate, and the soda ash phosphatide complex to free phosphatides. Upon settling or centrifuging, a minor portion or layer of aqueous material will separate, leaving a major portion or layer of a lecithin product comprising free oil, fatty acids and hydrated phosphatide. Most of the sodium sulfate and other inorganic salts will appear largely in the separated aqueous material. Any salts in the remaining lecithin product can be washed therefrom or can be permitted to remain therein as the small amount thereof is unobjectionable as a component of feeds.

Example: A soda ash soapstock containing 40% moisture and produced from the refining of a crude soya oil with soda ash was mixed with a 25% sulfuric acid solution in sufficient quantity to reduce the pH of the soapstock to approximately 5. Eight parts of the acid was re-





quired for 100 parts by weight (dry basis) of the soda ash soapstock. A fatty layer separated and was liquid at temperatures of 180° to 200°F. The fatty layer of lecithin product had a water content of 24%. After being dried in vacuo at 70°C., the dried product was a phosphatide concentrate, fluid at room temperature, containing 0.44% water, 2.11% phosphorus, 53.4% acetone insolubles and 66.8% TFA.

### Dry Additives Prepared from Soapstock

M.N. Pergament (U.S. Patent 3,051,571, August 28, 1962); has developed a process to provide a stock feed additive, such as a dry powdery substance having a fat content almost as high as the actual fat content of the feed material now being used, when the glycerin and moisture, impurities and unsaponifiable matters are taken into consideration, and which, because of its dry powdery nature, can be readily handled, stored over long periods of time and easily mixed with the stock feed to provide a homogeneous mixture.

Procedure: (1) An initial charge of 378 pounds of acidulated soapstock and/or fatty acids is pumped into a tank or kettle, provided with an agitator. (2) Seven gallons (or about 58 pounds) of water are then added to the charge in the kettle. (3) The mass is brought up to a temperature of about 190°F. while under constant agitation. (4) After sufficient agitation to insure fairly thorough mixing of the water with the initial charge, and while being heated to 190°F. as aforesaid, 50 pounds of quicklime is added. (5) Agitation is continued, and the temperature raised to about 230°F. while saponification begins to take place. The mass rises in the kettle, whereupon the rate of agitation is increased, and the mass settles to its original level. At this point, the rate of agitation is decreased and the mass again rises, whereupon the rate of agitation is increased until the mass again settles to its original level. With decreased agitation, the mass rises a third time (each rise being accompanied by the formation of clouds of steam); and a third time agitation is increased until the mass falls for the third and final time.

(6) The third, and final, agitation is continued for around four or five minutes at increased speed. (7) The entire batch is then quickly removed from the kettle and permitted to flow onto a concrete floor or into shallow large-area pans or the like. (8) The reaction between the quicklime, water, and saponifiable material continues to take place after removal from the kettle, being evidenced by small steam bubbles coming to the surface. (9) After cooling and drying, the mass is broken up, transported to the breaker or "hog", thence to the grinder or mill for final grinding.

### Soaps in Animal Feeds

The process of E.B. Patterson, R.E. Gray, and E.E. Rice (U.S. Patent 3,010,828; November 28, 1961; assigned to Swift and Company) provides a high fat content feed with good handling qualities. This involves adding salts of high molecular weight fatty acids (soaps) to animal feeds in order to provide a high energy product. It has been found that feeds having a soap content or a combined soap and fat content of 15% or more can be pelleted and will have a relatively lustrous, dust-free, and nonoily appearance. Sodium soaps have been found to be particularly advantageous for this purpose. Diet studies on chicks showed that soap was equal to tallow as an energy source, and at a level of 6.81% fat plus 4.0% tallow in feed, non toxic.



Example 1: One of the major deficiencies in feeds containing high levels of fat lies in the fact that they are almost impossible to pellet. In order to demonstrate one advantage gained in using soap as a substitute for other fatty materials, 5 lots of feed containing various combinations of soap and tallow were pelleted in a commercial feed-pelleting plant. Contrary to results obtained from other high fat content feeds, each of the lots below (200 lbs. per lot) pelleted without difficulty.

| Lot    | Soap,<br>percent | Tallow,<br>percent |
|--------|------------------|--------------------|
| 1..... | 3.41             | 7                  |
| 2..... | 4.64             | 6                  |
| 3..... | 5.68             | 5                  |
| 4..... | 6.81             | 4                  |
| 5..... | 2.84             | 5                  |

Example 2: The physical appearance of a feed is important in fixing its commercial value. In particular, a feed should have a high luster and a non-oily appearance and should be relatively dust-free. The below-listed feed lots were prepared by adding combinations of soap and tallow at a 10% level to a feed complete in every nutrient but fat.

| Lot    | Soap,<br>percent | Tallow,<br>percent |
|--------|------------------|--------------------|
| 1..... |                  | 10                 |
| 2..... | 1.15             | 9                  |
| 3..... | 2.27             | 8                  |
| 4..... | 3.41             | 7                  |
| 5..... | 4.64             | 6                  |
| 6..... | 5.68             | 5                  |
| 7..... | 6.81             | 4                  |
| 8..... | 7.95             | 3                  |
| 9..... | 11.36            |                    |

Although Lot 9 was somewhat dusty and light in color, Lots 3 through 8 were dust-free and had a non-oily appearance. These lots also had a relatively high luster. In contrast, Lots 1 and 2 were dark in color and felt greasy to the touch.

## HIGH FAT OILSEED MEALS

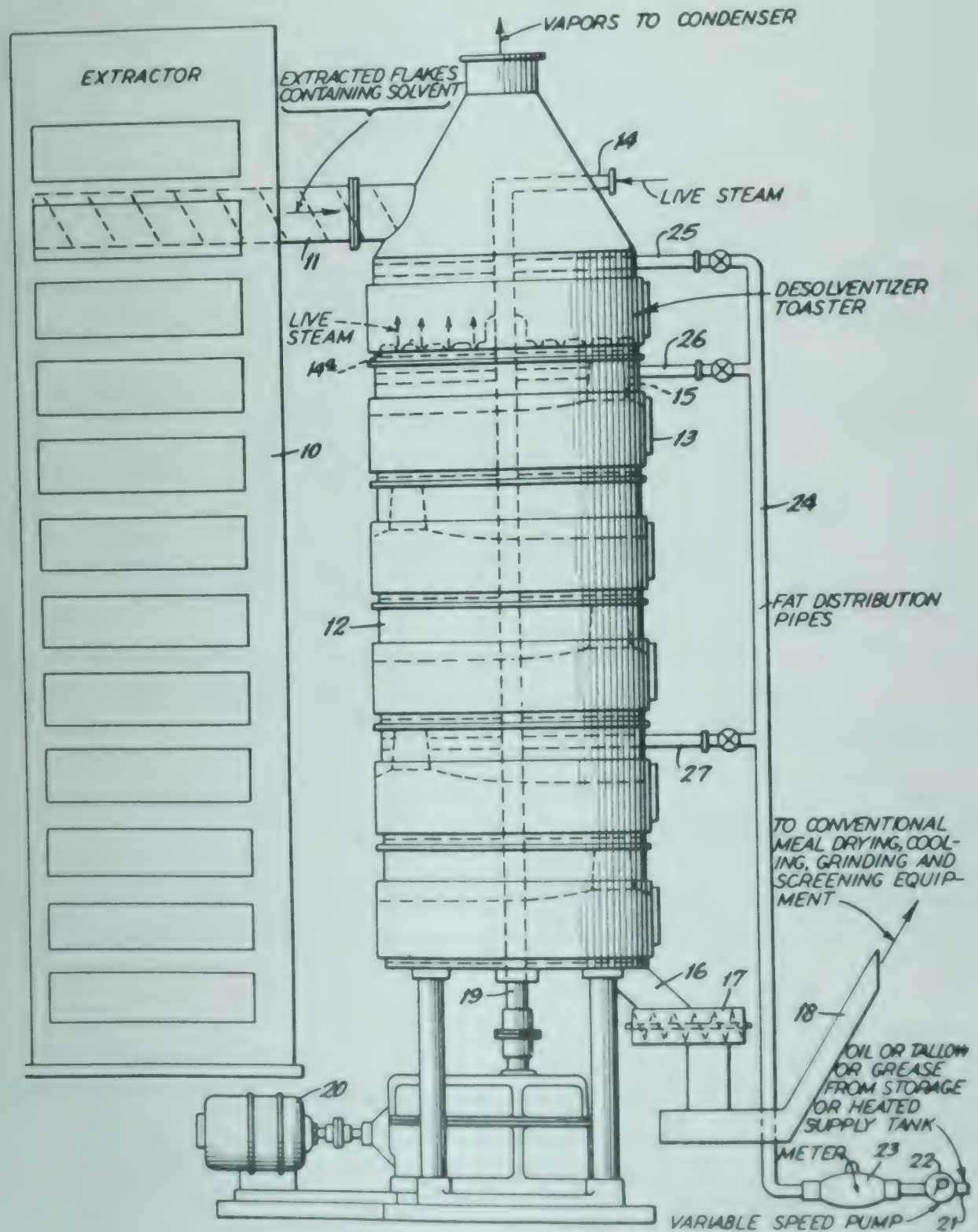
These processes relate to the production of oilseed meals. In addition to a high fat content with superior nutritional content these meals possess excellent handling and shipping properties.

### Dry Oilseed Meal

N.F. Kruse (U.S. Patent 2,928,738; March 15, 1960, assigned to Central Soya Company) has developed a process for preparing an oilseed meal having a high level of fat content with good keeping qualities while at the same time providing a dry texture meal having good flow and handling properties and no tendency to bleed into the container material. In the operation of the process embodiment illustrated in Figure 3.2 extracted soybean flakes containing 25 to 35% solvent are conveyed from the extractor 10 to the desolventizer-toaster or cooker 12 through the screw conveyor 11 while at the same time the fat or oil to be added is pumped

from storage or from a heated supply tank by the variable speed proportioning pump 22 and metered to the process by the flow meter 23. The fat is added to the extracted flakes, which are still wet with solvent, in the top kettle (No. 1) through the perforated distribution pipe 25. At the same time, the normal live steam addition by means of the sweep 14a in the top kettle 25. At the same time, the normal live steam addition by means of the sweep 14a in the top kettle is increased to furnish additional steam to heat the added fat.

FIGURE 3.2: HIGH FAT OILSEED MEAL



Source: N.F. Kruse; U.S. Patent 2,928,738; March 15, 1960



In this operation, the liquid solvent present in the flakes is effective in dissolving the fat, thus reducing its viscosity, and increasing its ability to penetrate into the meal particle. The extracted flakes at this stage are highly porous and readily absorb the fat. At the same time, the live steam acts not only to raise the temperature of the mixture but also, as it condenses upon the meal which is maintained below the boiling-point of water, furnishes a physical driving force which drives the fat into the interior of the meal particles. The subsequent drying of the meal operates in some manner which cannot be fully explained, to seal the fat within the meal particles. In effect, the fat becomes like the bound fat in the original soybean, and there is substantially no tendency for the oil thereafter to bleed during the packaging and shipping operations. The process is applicable with comparable results to other oilseed meals such as, for example, cottonseed meal, linseed meal, copra meal, corn oil meal, and the like.

### Thermal Pressure Preparation of High Fat Meal

In this process developed by N.F. Kruse (U.S. Patent 2,978,326; April 4, 1961; assigned to Central Soya Company, Inc.) mechanical energy is used to force the fat into the meal.

Example 1: Desolventized and toasted soybean meal was mixed with tallow at a rate of 4,000 lbs. of mix per hour, to bring the fat content to approximately 20%. 200 lbs. of steam per hour were added. The material was passed through a California pellet machine, using a thick 12/64 die. The oily mixture was formed in the pellet machine into hard pellets, which were cooled and then ground.

Example 2: Desolventized and toasted soybean meal was mixed with 12% of a blend of yellow grease and tallow and passed through an expeller fitted with a solid barrel, 150 lbs. of steam pressure being maintained on the jackets of the expeller tempering apparatus. The feed was in tempering for 3 3/4 minutes. The temperature of the caked product as extruded was 210° to 215°F. and the rate from the expeller was 1,350 lbs. per hour. In both instances the final product, after grinding, was dry and free flowing and it was found that the fat was bound into the meal, having no tendency to bleed into the paper or cloth containers. The process can be carried out with other oilseed meals, as corn germ meal and peanut meal.

### Oilseed Meal Dry Pellet

Hard dry feed pellets with a total fat content of up to 20% can be easily and successfully produced by adding the supplemental fat by spraying liquid fat onto hot pellets and mechanically agitating to evenly coat each pellet with fat in a process developed by E.J. Guidarelli (U.S. Patent 3,014,800; December 26, 1961; assigned to Cargill, Inc.). The oilseed meal used is a waste product from the extraction process contains some minor percentage of residual fat or oil. This residual fat content usually ranges less than about 3 to 4%. It is desired to add supplemental fat to increase the nutrient value of the feed and to improve its palatability as well.

Example: A copra feed mixture to be pelleted was introduced in a continuous stream into the mixing chamber of a 50 hp. California flat bed pellet mill and was there heated to about 115°F. and moistened to a total moisture content of about 15% (dry basis) by 100 psi



steam which condensed as it was blown into the feed mixture in the mixing chamber of the pellet mill. The heated and moistened feed mixture was discharged from the mixing chamber into the die of the pellet mill at a rate of about 3 tons per hour. The mixture was forced through the 3/16" holes in the die and cut into 3/8" lengths. The temperature of the pellets as they left the pellet mill was about 135°F. The pellets were then discharged directly into a 9" paddle conveyor 6' long and fitted at its upstream end with two 110° nozzles with a capacity of 0.3 gallon per minute. Liquified tallow was continuously supplied to a two gallon fat container fitted with a steam coil to maintain the fat at about 195°F. and maintained at 80 psi with compressed air in order to force the melted fat through the spray heads.

The tallow was sprayed onto the hot pellets in the conveyor as they passed beneath the spray nozzles. At the same time, the paddle conveyor agitated and tumbled the pellets to provide an even distribution of fat on the surface of the pellets. The conveyor was operated at a rate such that the coated pellets were subjected to the mixing and agitating process for a period of about 1 1/2 minutes during which time the fat was completely absorbed into the pellet resulting in a dry hard product. Upon discharge from the conveyor the pellets were immediately cooled. The total fat content of the resulting product was 9.8%. By practice of this process pelleted laying rations, turkey rations, and broiler rations have been made containing up to 20% fat by extending the absorption time to from 2 1/2 to 5 minutes. Hard dry copra meal pellets containing up to 15% to 20% fat have been successfully made by this process.

### PRODUCTS FROM CONDENSATE OF VEGETABLE OIL DEODORIZATION

#### Tocopherol Rich Stable Deodorizer Distillate

Tocopherols are found in natural fats and oils and particularly in vegetable oils such as cottonseed oil, soybean oil, safflower oil and the like. When such fats and oils are subjected to conventional deodorizer treatment, that is, blown with an inert gas such as steam, nitrogen and the like under vacuum, a by-product material usually separates from the inert gas in traps, condensers and the like. This by-product material is commonly referred to as deodorizer distillate. Invariably tocopherol components of the oil and fat being deodorized are found in deodorizer distillate and often at relatively high concentrations such as, for example, 2 to 15 weight percent. In this regard deodorizer distillates are rather complex mixtures comprising tocopherols, sterols, tocopherol esters, sterol esters, glycerides, and free higher fatty acids. Deodorizer distillates as obtained do not give satisfactory results when incorporated into the usual feeds and feed supplements. The reason is that under storage conditions and in the presence of minerals and other components of the usual feeds and feed supplements the tocopherols in the deodorizer distillates are not stable.

C.D. Robeson (U.S. Patent 3,212,901; October 19, 1965; assigned to Eastman Kodak Company) has developed a process for preparing a stabilized deodorizer distillate. This comprises admixing polyphosphoric acid with the deodorizer distillate and preferably heating the mixture for a short period of time. The resulting product is a stable deodorizer distillate, a stable, crude, tocopherol concentrate, suitable for animal and poultry feed supplements and feeds.

Example 1: This is a preparation of a stabilized deodorizer distillate as well as a feed



supplement containing the stabilized product. 42.5 grams of a commercial deodorizer distillate and 42.5 grams of a polyphosphoric acid were added to a beaker and the mixture heated on a steam bath for one hour with stirring. The reaction mixture was then mixed with ether (400 milliliters) and water (500 milliliters) whereupon a water phase and an ether phase formed. After removal of the aqueous phase, the ether solution was washed twice with water, and then slurried with a mixture of 20 grams of finely divided calcium silicate (marketed as Microcel E by Johns-Manville) and 60 grams of a commercial mineral mixture (marketed as Delamix by Limestone Products Corporation) while the ether was evaporated. This commercial mineral mixture comprised a number of mineral elements in a calcite flour carrier.

After evaporation of the ether the test product, a gray powder, was stored for 20 weeks in an open beaker at room temperature. A saponified extract of a sample of the product taken at the end of the storage period was assayed for total tocopherols by the Emmerie-Engel assay. The recovery of tocopherol was found thereby to be 84%.

A control product was made by admixing 20 grams of the same deodorizer distillate (but untreated with polyphosphoric acid) with 10 grams of finely divided calcium silicate (Microcel E), 50 grams of the same commercial mineral mix (Delamix) and ether to form a slurry. The ether was removed from the slurry whereby a gray powder product resulted. This product was placed in an open beaker at the same time as the test product and placed beside the test product beaker for 20 weeks. At the end of this time an Emmerie-Engel assay on a saponified extract of a sample of the product showed only 17% recovery of total tocopherols. Hence, treatment of the commercial deodorizer distillate with polyphosphoric acid resulted in an increased stability of the tocopherols content when exposed to air in the presence of minerals.

### Vegetable Oil, Animal Fat, Crude Well Oil Composition

A process of A. Rosenberg (U.S. Patent 2,835,584; May 20, 1958;) provides feed manufacturers and raisers of farm animals with two types of fatty materials which not only constitute a concentrated source of energy and control dustiness in feeds, but in combination impart to the feeds all the nutritional advantages inherent in fats, to wit, provide an excellent source of the essential fatty acids, provide an excellent stabilizing vehicular material for the fat-soluble vitamins A, D, E and K, provide an excellent source of vitamin E, and further provide an excellent medium for promoting absorption of the fat-soluble vitamins from the gastro-intestinal tract. Lowcost by-products of edible oil industries are used as the source of the basic components, such as acid oil, the source of essential fatty acids, which is the material obtained by the acidulation of the soap stock that accumulates in the alkali refining of vegetable oils. The acid oil, drawn off the surface of the acidified mixture, is liquid at room temperature, bitter in flavor and dark brown to black in color. The other type of fat used is hydrogenated animal fat of feed grade quality in which there has been dissolved "hot well oil".

"Hot well oil" is the water-insoluble portion of the condensate obtained in the deodorization of vegetable oils by high-vacuum steam-distillation and is a source of Vitamin E. The unmodified hot-well oil has true vitamin E activity of about 2,000 international units per 100 grams, equivalent to about 1.5 g. % as d-alpha-tocopherol-acetate. The emulsifier, when added, consisted of 3% commercial lecithin derived from soya bean oil. The fatty beads were made by heating the hydrogenated tallow to a temperature of about 10°C. above its melting



point, the lecithin was added, and also the hot well oil, when used, added thereto. The heated solution of the hydrogenated tallow with the additives was spray-chilled to yield small beads predominantly 20 to 60 mesh in size. Hot well-oil concentration in the hydrogenated animal fat will vary from as little as 0.2% to as much as 24%, dependent upon the levels of the hydrogenated animal fat and vitamin E desired in the ration.

The hydrogenated animal fat of melting point above 52°C., and containing hot-well oil and emulsifier, is added as discrete particles in concentrations of from 1 to 5%. The ratio of one type of fat to the other (added oil on the one hand and hydrogenated tallow on the other) in the aforesaid combination (required for imparting to feeds all the nutritional advantages inherent in fats) may vary from 1:5 to 5:1 (acid oil: hydrogenated animal fat), with the total fat supplement ranging from 2 to 10% of the feed. The rations were prepared by spraying them with 3% soya bean acid oil in cases where the acid oil was added. The beads of the hydrogenated tallow were mixed with the sprayed rations, in the desired percentage by distributing them uniformly in the rations. Farm animals ordinarily do not ingest hydrogenated fats of melting points above 52°C. Fat (not physiologically available) which is unabsorbed by chickens, for example, is found in the excreta. Results of diet study are shown below.

*Physiological availability to the chicken<sup>1</sup> of the fat in poultry rations*

| Ration                  |     | Extra Fat Supplement  | Fat Present in Ration, Percent | Analyses of Droppings         |              |                             |                               |              |                             |
|-------------------------|-----|---|--------------------------------|-------------------------------|--------------|-----------------------------|-------------------------------|--------------|-----------------------------|
|                         |     |   |                                | Birds—7 days old              |              |                             | Birds—70 days old             |              |                             |
| Type                    | No. |   |                                | Total Solids (T. S.), Percent | Fat, Percent | Fat on T. S. Basis, Percent | Total Solids (T. S.), Percent | Fat, Percent | Fat on T. S. Basis, Percent |
| Commercial <sup>2</sup> | 1   | None  | 3.2                            | 70.8                          | 2.04         | 2.88                        | 25.3                          | 0.82         | 3.24                        |
|                         | 2   | 5% Tallow, M. P.=45° C.   | 8.2                            | 66.3                          | 1.97         | 2.97                        | 27.8                          | 0.84         | 3.32                        |
|                         | 3   | 5% Hydrogenated tallow beads, M. P.=58° C.  | 8.2                            | 64.3                          | 2.38         | 3.70                        | 23.3                          | 0.95         | 4.07                        |
|                         | 4   | 5% Hydrogenated tallow beads, M. P.=58° C; beads containing 5% hot-well oil and 3% lecithin.                      | 8.2                            | 69.6                          | 2.01         | 2.89                        | 24.3                          | 0.76         | 3.12                        |
|                         | 5   | None  | 0.0                            | 68.7                          | 1.66         | 2.42                        | No survivors.                 |              |                             |
|                         | 6   | 5% Tallow, M. P.=45° C.   | 5.0                            | 67.3                          | 2.62         | 3.90                        | No survivors.                 |              |                             |
|                         | 7   | 5% Hydrogenated Tallow beads, M. P.=58° C.  | 5.0                            | 68.2                          | 3.08         | 4.52                        | No survivors.                 |              |                             |
|                         | 8   | 5% Hydrogenated tallow beads, M. P.=58° C; beads containing 5% hot-well oil and 3% lecithin.                      | 5.0                            | 66.0                          | 2.61         | 3.96                        | No survivors.                 |              |                             |
| Fat-Free <sup>3</sup>   | 9   | 3% Soybean acid oil.  | 3.0                            | 70.3                          | 1.84         | 2.62                        | 28.2                          | 0.77         | 2.73                        |
|                         | 10  | 3% Soybean acid oil+5% tallow, M. P.=45° C.   | 8.0                            | 73.2                          | 1.98         | 2.71                        | 22.4                          | 0.66         | 2.94                        |
|                         | 11  | 3% Soybean acid oil+5% hydrogenated tallow beads, M. P.=58° C.  | 8.0                            | 68.4                          | 2.61         | 3.82                        | 26.4                          | 1.06         | 4.02                        |
|                         | 12  | 3% Soybean acid oil+5% hydrogenated tallow beads, M. P.=58° C.; beads containing 5% hot-well oil and 3% lecithin. | 8.0                            | 67.5                          | 1.78         | 2.64                        | 25.6                          | 0.71         | 2.77                        |

<sup>1</sup> 20 one day old White Rock chicks (Robert's Strain) in a group.

<sup>2</sup> Balanced commercial ration of 24 percent protein content considered to be completely adequate for growing chick.

<sup>3</sup> Basal fat-free ration as described by Reiser, Journal of Nutrition, vol. 42, p. 319 (1950).

The completely hydrogenated tallow particles, containing hot-well oil and an edible emulsifying agent (lecithin) are as readily digested and absorbed as unhydrogenated tallow sprayed on the commercial feed. When either of those fat supplements is added to the commercial ration containing about 3% of residual vegetable oil, complete absorption is obtained (no increase of fat in the droppings). Completely hydrogenated tallow is utilized to a lesser extent. All these fat supplements are utilized less efficiently when added to the fat-free ration (increased excretion of fat, calculated on a total solids basis over the endogenous excretion, that are noted when no fat supplements are present in the ration). However, when acid oil in 3% concentration is added to the fat-free ration, effective utilization of the



completely hydrogenated tallow containing hot well oil and lecithin is noted. The hydrogenated tallow alone is not as effectively utilized under these conditions.

### Powdered Absorbent Feed Materials Containing Crude Hot Well Oil

Crude hot well oil is the water-insoluble portion of the condensate obtained in the deodorization of vegetable oil by high-vacuum steam distillation. It represents a tremendous concentration of the objectionable odorous materials that develop in vegetable oils when separated from their natural environment. Moreover, these components are further modified at the high temperatures of steam deodorization, viz., 430° to 460°F., and these changes are accelerated by the high concentration of the pro-oxidant iron in the crude hot well oil. The objectionable flavor of crude hot well oil and its high iron concentration have been responsible for feed manufacturers refusing to add this crude material to feeds. It was feared that the hot well oil additive might not only interfere with feed consumption, but that the flavor might carry through into the tissues of the animals.

In this process, A. Rosenberg (U.S. Patent 3,015,563; January 2, 1962; assigned to Commercial Solvents Corporation) found that tests conducted on chickens subsisting on diets containing up to 3% of hot well oil indicated that no adverse flavors were imparted to the tissues when the tissues of the test and control birds (on the same ration but without the added crude hot well oil) were cooked and subjected to a flavor panel. Hot well oil up to 5% of the ration was still acceptable. Raw unprocessed hot well oil does provide a unique source of stable Vitamin E and plant sterols which may be made available in a variety of ways. The raw unprocessed hot well oil can be employed by spraying or otherwise distributing it over dry feed ingredients, such as solvent-extracted soybean meal, powdered corn cobs, powdered citrus pulp, skim milk powder, solvent-extracted cottonseed meal, etc., in an amount of up to 35%.

An excess of the raw unprocessed hot well oil renders the product oily whereby it loses its free flowing characteristics. The tocopherol and vitamin E content, even when more than 35% is absorbed by the feed ingredients, exhibits full retention. The raw unprocessed hot well oil may be embodied in discrete particles (beads or flakes) of fat where the vehicular material has a melting point of 50°C. or higher. In such beads the content of the raw unprocessed hot well oil may go as high as 15% with retention of the free-flowing characteristics of the beads; with vehicular material of melting point of 58°C. or higher, the concentration of the crude hot well oil may be as high as 40%. "Microcel", a synthetic calcium silicate, can absorb up to twice its weight of raw unprocessed hot well oil without losing its dry free-flowing characteristics. Antioxidants can be included.

Example: A product was made by spraying 270 parts of raw unprocessed hot well oil; this has a vitamin E potency of 5,000 international units of vitamin E per pound) on 630 parts of solvent-extracted soy flour in a mixer. This product contained 30% of raw unprocessed hot well oil and provided 7.8% of plant sterols. Its vitamin E content was 1,600 international units per pound of the mixture, according to the chick bioassay. Full vitamin E content was retained after 6 weeks' storage at room temperature; after 10 weeks' storage vitamin E retention was still 70%. The addition of one and one-half pounds of the freshly-prepared mixture to one ton of feed provided 2,400 units of Vitamin E per ton, regarded by some authorities as an effective practical supplement of feeds containing some residual native vitamin E.



This product was also added in the amount of ten pounds per ton of feed. The product provided 35 grams of plant sterols per pound. The feed contained also 5% of beef tallow; the tallow containing 200 milligrams percent of cholesterol provided about 90 grams of cholesterol per ton of feed. This cholesterol was readily neutralized by the 10 pounds of the product of this example per ton of feed. The ratio of plant sterols to cholesterol in the ration was almost 4:1, well in excess of that required to "tie-up" all dietary cholesterol and prevent its absorption from the digestive tract. The vitamin E contribution from the 10 pound supplement was 16,000 units per ton of feed regarded by some authorities as desirable when no reliance can be placed upon the native vitamin E once present in the feed components.

### VEGETABLE OIL EMULSIONS

#### Vitamin Enriched Vegetable Oil Emulsion

G.D. Elenbogen and M.A. Clovis (U.S. Patent 3,438,782; April 15, 1969; assigned to Vitamins, Inc.) has developed a process for a vegetable oil base nutritive supplement in the form of an emulsion which is especially suitable for carnivorous fur bearing animals (mink, fox, etc.). The vegetable oils employed may be any of the edible vegetable oils (saturated and unsaturated oils) as wheat germ oil, soybean oil, corn oil, safflower oil and the like. The polyunsaturated vegetable oils are particularly desirable. In addition, the feed supplement is further enriched by the addition of a wide variety of oil soluble vitamins, for example, vitamins A, D<sub>2</sub> and E and water soluble vitamins as vitamins B<sub>1</sub>, B<sub>2</sub> and C. The vitamins are dissolved in the appropriate phase of the supplement, and therefore uniformly dispersed.

It is preferred to employ an oil content of from about 60 to 70% by weight, in the supplement. The fat is emulsified with a suitable emulsifying chemical agent, as for example, the sorbitan esters of fatty acids, polyoxyethylene sorbitan esters of fatty acids and mixed fatty acids. Also, the monoglycerides and polyglycerides, soap stearates and higher fatty alcohol emulsifying agents can be used. A mixture of polyoxyethylene sorbitan monooleate and polyglycerol oleate is especially desirable for this keeps the emulsion clear which is desirable for feeding purposes. 30 to 50% by weight emulsifying chemical agent is preferred. A small quantity of water (1.5 to 6%) is employed in the oil supplement in order to render it possible to incorporate water-soluble vitamins into the supplement.

Example: In preparing the fat supplement, the water-soluble vitamins are admixed with the water. The oil-soluble vitamins are admixed with the oil, and the emulsifying agent is then added to the mixture. The material is thoroughly admixed by any suitable mechanical mixer or blender such as a Waring Blender or other impeller type blenders. The oil supplement formed in accordance with this process is a water in oil emulsion, and the oil is the external phase. The rancher further dilutes the oil supplement with water just prior to incorporating it into the feed ration, and the emulsion inverts to an oil in water emulsion. The quantity of oil supplement then incorporated into the feed ration depends upon the particular nutritional requirements for the animal.

#### Vegetable Oil-Lecithin-Tocopherol Composition

The process of S.J. Golub and N.E. Silbert (U.S. Patent 3,117,866; January 14, 1964;



assigned to Activator, Inc.) concerns the preparation of an orally ingestible composition, for an animal feed supplement, comprising a vegetable or animal oil having material content of unsaturates and containing substantial amounts of additive lecithin (optimum 6 to 8%) as well as tocopherols (optimum 0.5 to 0.6%).

**FORMULATION - A:** Concentrate product (approximately 53.5 lbs.). Step 1: Dissolve 18.5 g. of 2,6-di-tert-butyl-4-methyl-phenol (such as Ionol USP of Shell Chemical Corp.) in 10 lbs. of wheat germ oil. Heat until dissolved, at 65° to 70°C. for about 10 to 12 minutes, stirring continuously. Add 28.25 lbs. refined lecithin. Heat slowly to the optimum temperature of 85°C., maintaining the same for approximately 5 minutes while stirring and then cool slowly. The critical temperature range which is applicable to this heating comprises approximately 80° to 90°C.

Step 2: In a separate container, mix 18.5 grams of powdered propyl gallate and 18.5 grams of propylene glycol. Heat gently, stirring until the propyl gallate is fully dissolved, the solution revealing a tawny port wine color.

Step 3: Separately add 1.5 lbs. each of brain and liver lipids to 10 lbs. of wheat germ oil. Heat to 85°C., with continuous agitation, maintaining the temperature for 5 to 10 minutes. The temperature may be in the aforesaid range of 80° to 90°C., 85°C being optimal. During slow agitation, mix the resultant solutions of step 1, step 2, and step 3, preferably in that order.

Step 4: To the combinative solution of steps 1, 2 and 3, add 1 kilo of mixed tocopherols (such as type 4-50 Eastman or the like). Then add 19.25 cc. of a mixed vitamin A and D, (Myvax of Distillation Products Co. being satisfactory). The over-all mixture is re-heated to the optimum temperature of 85°C. for five minutes, cooled and, if desired, filtered to remove particulate brain and liver solids, although this latter expedient is not necessary. As designated above, the temperature utilized in this critical heat treatment may be within the approximate range of 80° to 90°C.

**FORMULATION - B:** The final composition (approximately 413.5 lbs.). To 260 lbs. of soy bean oil (or other oil to be utilized) add the 53.5 lbs. of the concentrate product of A. During continuous agitation, add to the latter solution 100 lbs. of wheat germ oil (preferably containing 2 international units of vitamin E and having zero peroxide value). Again desirably raise the temperature of the ultimate mixture to 85°C., terminate the heating, and cool under atmospheric conditions with stirring for approximately 10 minutes. In further reference to the combinative aspects of the ingredients as they are coordinated in step 1 of this formulation, the phenol component is a commercially available antioxidant, and it is initially admixed with the designated quantity of wheat germ oil, a relatively small proportion of that in the ultimate composition, for the purpose of effecting an initial stabilization during the preliminary stages of preparing the product, especially in view of the attendant heat treatment of the said wheat germ oil in the temperature range of 80° to 90°C. Thus the designated phenol is intended to afford a protective function with respect to the possibility of oxidative effects on the oil during the applicable heating, and may likewise comprise a stabilizer with respect to any oxidation of the lecithin which is likewise supplied in this initial step.

## Fats and Oils

The following is indicative of the desirably high unsaturation characteristics of the optimum product comprising Formulation A and B.

|                              | Percent |
|------------------------------|---------|
| Oleic acid.....              | 21.13   |
| Linoleic acid.....           | 49.91   |
| Linolenic acid.....          | 4.59    |
| Cephalin acids.....          | 3.90    |
| Arachidonic acids.....       | 0.161   |
| Other unsaturated acids..... | 0.118   |

Biological effects afforded by use as a feed supplement for poultry is illustrated by the following tabulation.

| Groups             | Avg. Terminal<br>wt. Before<br>Sacrifice,<br>lbs. | Amt. Grain<br>Consumed per<br>lb. Poultry<br>Produced, lbs. | Percent<br>Savings of<br>Feed |
|--------------------|---|---|-------------------------------|
| 3 (200 birds)..... | 3.385   | 2.998   | Control                       |
| 1 (100 birds)..... | 3.45  | 2.706   | 9.8                           |
| 2 (30 birds).....  | 3.483   | 2.653   | 11.5                          |
| 4 (400 birds)..... | 3.40  | 2.600   | 13.3                          |

The tests commenced with chicks that were one day old. Pen 3 comprised the control group, and was supplied merely with a standard commercial poultry feed and pure drinking water. With respect to pen 1, the same poultry feed was provided as in pen 3, but the drinking fluid comprised a 1% aqueous emulsion of the Formulation, constantly available during the ten week test, conforming with the life of the birds.

As for pen 2, the chicks of this group received pure drinking water throughout their life span. Moreover during the initial five weeks, the chicks of this pen were supplied with the aforesaid standard commercial poultry feed, embodying 1% by weight of the Formulation. Concerning pen 4, the feed was restricted to the aforesaid commercial type likewise containing 1% by weight of the Formulation. The liquid administered to the chicks of this pen was pure drinking water. Thus the chicks of this fourth group were recipients of the fortified feed during their entire ten weeks of life, as distinguished from the five week interval in which the birds of pen 2 were administered the same fortified feed.

### FATS IN FEEDS

Fats improve the nutritional value of animal rations, and are hence being widely used to-day as components of animal feeds. In the poultry-raising industry, it has become the practice to supplement poultry feed with varying amounts of vitamins, minerals, antibiotics and fat, to obtain maximum growth in as short a time as possible, as well as to obtain a high ratio of feed conversion, i.e., the greatest weight per pound of feed consumed. Such feeds are known as "high-energy" poultry feeds and may contain up to 10% or more by weight of added fat. High-energy, fat-containing feeds may also be used as rations for laying hens, as well as for other livestock.



### Digestible, Discrete, Fat Flakes

Hydrogenated fats of high-melting point which offer the conveniences of uniform distributability in packaging and handling are not used because of the fact that the livestock has extreme difficulty in digesting such fats.

The process of A. Rosenberg (U.S. Patent 3,011,892, December 5, 1961; assigned to Commercial Solvents Corporation) provides a fat in particulate form based on hydrogenated fat, for easy mixing with other feed components, the fat being readily available to the animal for nutritional purposes.

The fat product of this process is characterized by the fact that it is a blend of: (1) one or more high-melting fats obtained by completely or almost completely hydrogenating the fat, with (2) one or more liquid oils that have not been hydrogenated. The high-melting fat component of this described fat product may be a highly hydrogenated vegetable oil (such as, cottonseed, soybean, peanut or corn oils, etc.), a highly hydrogenated animal fat or oil (land or marine), such as lard, tallow, sardine, menhaden, cod liver, pilchard, halibut, etc. The high-melting component of the novel fat product of this invention has a melting point of about 54° to 70°C. and an iodine value of about 0 to 6; and the fatty acid radicals thereof are present in a random distribution.

The high-melting component of the fat product of this invention comprises about 65 to 90% of the product. The remainder (35 to 10%) comprises the non-hydrogenated liquid oil. These oils may be the unhydrogenated, vegetable oils (such as peanut, cottonseed, corn, soybean, linseed, etc.) or the marine oils (such as, sardine, menhaden, cod liver or halibut, etc.). The liquid oil component of this already described product has an iodine value of about 90 to 200 and the fatty acid radicals thereof are in a non-random or random distribution. The ratios of fat in the blend are critical as seen in the following example.

Example: To 70 parts of molten, almost completely hydrogenated soybean oil with a melting point of 68°C. and an iodine value of 2 are added 30 parts of menhaden oil of 160 iodine value. The resulting blend has an iodine value of 49 and a melting point of 60°C., and the fatty acids are present in non-random distribution in the triglycerides. The blend is heated to a temperature of 75°C. and the fat stream passed over a chill-roll to provide discrete fat flakes of less than 1 mm. in thickness and less than 5 mm. in diameter. On biological assay for digestibility, the fat is found to be absorbed to the extent of 82%.

A 70:30 blend of limpid soybean oil and menhaden oil hydrogenated to an iodine value of 49 has the same coefficient of digestibility but fails to yield satisfactory discrete fat flakes since the fat has a melting point of only 49°C. A 70:30 blend of limpid soybean oil and menhaden hydrogenated to an iodine value of 12 has a melting point of 61°C. This fat yields discrete fat flakes but these flakes are unacceptable since the fat is digested to the extent of only 35%.

### Choline-Fat Composition

The inclusion of added fat in a poultry feed has made it necessary also to increase the amount of choline in the feed. Choline is a member of the B-complex group of vitamins, and one

## Fats and Oils

of its functions nutritionally is concerned with the metabolism and transportation of fat. E. Lorz (U.S. Patent 2,970,911, February 7, 1961; assigned to Hoffman-Taff, Inc.) has a process for preparing a non-hygroscopic, fat supplement for incorporation in an animal or poultry feed in which choline salts are in stable and homogeneous dispersion in proper proportions with respect to the fat. The choline compound reacts with the free fatty acids in a fat so that the resulting choline salts of fatty acids are intimately dispersed throughout the fat.

Example: 200 pounds of feed-grade fat containing about 6% free fatty acids are placed in a suitable vessel provided with a mechanical agitator and heating device. The vessel is heated until fat becomes liquid (usually at a temperature between 32° and 104°F.). After the fat is liquified, the agitator is started and a solution of 4 1/2 pounds of choline base in a minimum quantity of water is slowly added to the stirred fat. The stirring is continued while heating the mixture over a period of from 2 to 4 hours at a temperature of from 160° to 200°F. to complete the reaction. The mixture, at a temperature of about 180°F., is then sprayed directly onto dry, finished poultry feed in a suitable amount to provide the fat content desired. Under such conditions, the fat-choline composition is uniformly distributed in the feed, which may now be shipped to the consumer.

Feed prepared in this manner contains approximately 0.02 pound of added choline—calculated as choline base—per pound of added fat. For example 50 pounds of the above fat-choline mixture may be added to one ton of feed, each pound of feed thereby containing 2.5% of fat and each pound of feed containing 227 milligrams of added choline.

### High Fat Content Food Pellets

G.T. Lanz (U.S. Patent 2,945,764, July 19, 1960; assigned to Ralston Purina Company) has a process for making stable pellets of high fat content. The pellets are formed and dried to remove a substantial amount of moisture at least at or near the surface. While hot and tumbling, they are sprayed with hot liquid fat. Poultry food pellets so produced, according to standard tests are 300% harder for a 10% by weight fat content than pellets made by other processes having only a 4% fat content.



## MOLASSES AND FLAVORINGS

### MOLASSES

Molasses is one of the most common products used in the preparation of food supplements for livestock. Blackstrap molasses is relatively inexpensive, is a natural food for animals, and has an appealing taste. Unfortunately at ordinary temperature molasses is a liquid which is both very viscous and highly hygroscopic. This hygroscopic property makes it hard to handle in dry or pelleted feeds. These processes offer improved methods for using and handling molasses.

### Non-Hygroscopic Molasses Product

R.M. Winn (U.S. Patent 3,033,684; May 8, 1962; assigned to Blackstrap Dry, Inc.) has a process to provide a dry, non-hygroscopic form of blackstrap molasses which is accomplished by adding small amount of tapioca or tapioca and saponin. Of the two additives, tapioca is the one responsible for rendering the mixture non-hygroscopic. This phenomenon appears to be due to the coating of each particle of the mixture with a fine film or coating of tapioca.

Saponin has two very beneficial effects. For one, it accelerates the drying during mixing. Its other effect is a synergistic action, judged to be due to a breaking up of the gums in the molasses, which greatly increases the protein content of the final mixture relative to the starting materials, typical increases ranging from around 40% to over 100%.

The livestock feed of this process in its preferred molasses-high protein form, may be produced or formed by thoroughly mixing in a suitable mixer to the point of dryness, underhydrated molasses and a high protein substance in the presence of quantities of tapioca and saponin so small relative to the total mixture as to add but little to the cost of the raw materials. If only tapioca is added, its quantity by weight should be around 1% of the total mixture. However, by adding saponin up to the ordinarily sufficient maximum of around 0.008% by weight of the total mixture, the quantity of the tapioca required for the non-hygroscopic product can be reduced to somewhat less than 0.4% at which its ratio to the saponin will be about 50:1.

Example: A non-hygroscopic molasses-high protein feed may be formed from the following mixture.

## Molasses and Flavorings

|                     | <u>Parts by Weight</u> |
|---------------------|------------------------|
| Blackstrap molasses | 560                    |
| Soybean meal        | 140                    |
| Tapioca             | 2.5                    |
| Saponin (pure)      | 0.05                   |

### Dried Molasses Product

A process for making a dehydrated, noncaking molasses product has been developed by B. Kviesitis (U.S. Patent 2,967,106; January 3, 1961; assigned to Vy Lactos Laboratories, Inc.) which involves mixing dehydrated molasses with a wetting agent, especially sorbitol.

Blackstrap molasses is composed of a solution primarily of sugars and organic acids and minerals in which some colloidal substances, such as gum and insoluble minerals, are suspended. Particles of this colloidal substance are of different sizes, thus presenting problems in dehydration process. Success of drying depends on uniform atomization of liquid material. Therefore, molasses should be purified from all colloidal parts which are interfering in proper atomization. Generally, molasses is sold of density ranging from 79.5° to 90° Brix. Purification of this molasses is accomplished by the following method:

- (1) Molasses diluted with water to 42° Brix.
- (2) Heated to 90° to 100°F.
- (3) One percent by weight of calcium hydroxide added and the mixture agitated for ten minutes.
- (4) Mixture is left in a vertical tank with conical bottom for twelve to twenty-four hours to allow to settle suspended colloidal particles. The clear supernatant liquid is then transferred to operating tanks. This purified liquid is ready for dehydration.

The diluted molasses or like, after purification, is then first heated to approximately 190°F. and the final drying is usually accomplished by hot air at temperatures of approximately 300° to 400°F. The result is a dry powder and, in order that it will continue as such through the rest of the processes, including packaging, approximately one percent by weight of tri-calcium phosphate is injected into the powdered material. Before packaging a wetting agent is added so that the final product will have lyophilic nature and good blending and working characteristics.

Example: Lecithin at the rate of two parts by weight is added to 98 parts by weight of spray dried molasses powder and mixed in conventional ball mill or any other suitable blending equipment until uniform material is obtained. This product can be modified to have a more free flowing character, in this case 0.5 or 1.0% by weight of noncaking compound, such as tricalcium phosphate, is added and mixed in a conventional Ribbon or any other mixer.

### Molasses on an Oat Hulls Carrier

In another process, B. Kviesitis and W.E. Rogerson (U.S. Patent 3,395,019; July 30, 1968;



assigned to Vy Lactos Laboratories, Inc.) describe a method to treat and/or process oat hulls so that they will successfully absorb and retain such materials as molasses, fish solubles, and the like.

Example: A quality of oat hulls was ground to a particle size of less than Tyler mesh No. 14. Water was then added to the ground oat hulls at a ratio of 60% by weight. The ground oat hull-water mixture was then mixed in a conventional ribbon type mixer for approximately 10 to 20 minutes. The temperature of the mixture was then adjusted to approximately 75°F. A yeast starter was prepared by mixing one pound of compressed yeast of Saccharomyces cerevisiae with two quarts of water and one pint of cane molasses and was propagated for four hours at approximately 80°F. The yeast starter was then added to the oat hull-water mixture at the ratio of one pound of compressed yeast to 500 pounds of oat hulls and thoroughly mixed.

This mixture was maintained at a temperature of 80°F. for approximately one hour (preferred range of temperature being 70° to 85°F. with best results being obtained at 80°F.). The mixture was then baked and dried in an ordinary rotary kiln at a temperature of 250° to 300°F. for a period of 30 to 45 minutes. The yeast is deactivated by the baking and drying. However, prior to such baking and drying, the yeast changes or modifies the oat hulls in several ways, among them being: (1) the formation of alcohol and carbonate dioxide; (2) the production of soluble carbohydrates such as sugars and dextrans from insoluble forms of starch; (3) the production of various organic acids such as lactic, acetic and at times butyric; (4) a partial solution of the protein compounds; and (5) the formation of amide and ammonium compounds from insoluble protein.

All of these changes are due mainly to the enzymes produced by the yeast. All of these changes improve the absorption capacity of the oat hulls and improve the palatability of the finished product prepared from treated oat hulls and cane molasses. Enzymes may be substituted for the yeast and will produce changes 2 to 5 above.

The next step in the process is to mix the treated oat hulls with water and molasses. The amount of water and molasses added to an amount of baked oat hulls depends upon the desired sugar content of the finished product. For example, 1,200 pounds of feed grade cane molasses may be added to 500 pounds of water and the resultant mixture added to approximately 1,160 pounds of baked oat hulls. The final product will be approximately 2,000 pounds of dried finished product having a sugar content of approximately 29.0%.

As a further example, 1,800 pounds of feed grade molasses may be mixed with 700 pounds of water and the resultant mixture added to 7,450 pounds of baked oat hulls. The final product will be approximately 2,000 pounds of dried finished product having a sugar content of approximately 43.0%. The best finished product will be achieved when the water and molasses are mixed together and heated to approximately 180°F. before they are introduced to the treated oat hulls. The next step in the process is the mixing of the mass by any convenient method for a period of time (usually 20 minutes) to permit the successful absorption of the molasses or like into the oat hulls.

The last step is the placement of the mixed mass into a drying means such as a rotary drier. In the drying method used, hot air having a temperature of 500° to 700°F. was introduced



in the input end of the drier. The outlet temperature of the air was approximately 240° to 300°F. The mixed mass usually requires 30 to 60 minutes of drying time. The final finished product emerges from the rotary drier with a temperature of below 140°F. and a moisture content of 0.5 to 5.0%. The finished product coming from a drier will not be of a sticky nature, but may be easily handled, packaged, stored, transported, and mixed with other stock feed. Although molasses has been mainly referred to, obviously fish press water, fish solubles and the like may be substituted for the molasses when desired.

### Molasses-Vermiculite Product

J.A. Kelley (U.S. Patent 3,284,209; November 8, 1966; assigned to W.R. Grace & Co.) describes a process for a new form of vermiculite which can be produced by compressing exfoliated vermiculite into blocks using the techniques and equipment utilized in forming salt blocks; or by extruding compressed exfoliated vermiculite through a die; or by forming a sheet or film as by passing the exfoliated vermiculite between rollers; or by stamping pellets, tablets or "slugs" of compressed expanded vermiculite.

Example — Production of Blackstrap Molasses Vermiculite Product: 150 pounds of properly expanded vermiculite of dominant screen fractions of 16 to 40 mesh particles will readily absorb a hot mixture (approximately 250°F.) of 350 pounds of blackstrap molasses and 50 pounds of water and permit its subsequent drying in a rotary dryer when operating such dryer so as to produce a dry composition containing approximately 60% dry molasses solids at a discharge temperature of 170° to 180°F.

Additional molasses solids can be dried on this vermiculite-molasses product by appropriately recycling with subsequent hot molasses additions through the same dryer to result in a product containing up to 80% dry molasses solids. Either blackstrap molasses vermiculite product may then be compressed at a pressure of 16,000 psi, for example, to form a relatively firm, hard, non-hygroscopic product which upon immersion in water will leach out the molasses and in the process cause the contained vermiculite carriers to reexpand and thus hasten the disintegration of such immersed completed blocks in water.

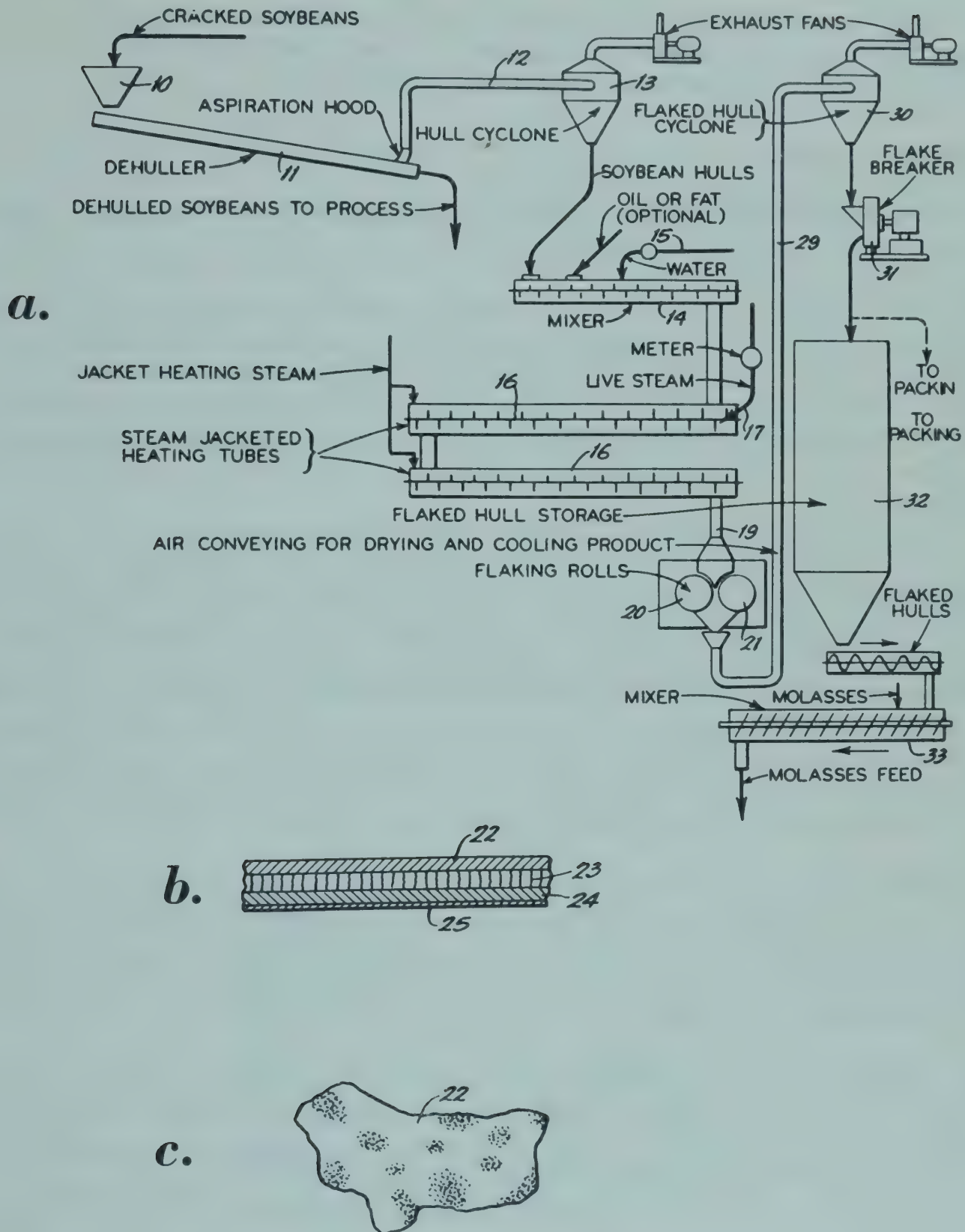
### Fibrous Absorbent Flake from Hulls

N.F. Kruse (U.S. Patent 2,952,540; September 13, 1960; assigned to Central Soya Co., Inc.) has a process to convert hulls and other high fiber materials into a material of low bulk density having a high capacity for absorbing liquid feed ingredients, the resulting feed material, after absorbing such liquids, presenting outer dry surfaces and being free flowing. For the purpose of simplicity, the process herein will be described in connection with a soybean hull, it being understood that the process may be similarly employed with other types of hulls and high fiber materials.

The cracked soybeans (see Figure 4.1a) are passed from a bin 10 into a dehuller 11 and the removed hulls are then drawn through pipe 12 into the hull cyclone 13. The hulls are passed from the cyclone to a mixer 14 in which water is added at 15. The moistened hulls then pass into a heat-jacketed conduit 16 and into which live steam is introduced at 17. The addition of enough water to bring the moisture content to 15 to 25% is highly satisfactory, best results being obtained when the moisture content after the addition of live steam



FIGURE 4.1: FIBROUS ABSORBENT FLAKE FROM HULLS



reaches 18 to 20%. From 10 to 15% by weight of steam is found to be sufficient, since this tends to give a product temperature of 190° to 210°F. Steam within the heated conduit tends to dissolve the wax coating of the hulls while at the same time making the material plastic. The rapid heating also destroys the urease or other undesirable enzyme activity which may be present. The steam condenses upon the hulls and thus meters moisture thereon so as to evenly coat the hulls with moisture. As a result of the steam and heat application, it is found that the hulls tend to curl into a tubular or roll shape.

The curled hulls pass from the conduit 16 through the pipe 19 and thence through the flaking rolls 20 and 21. One of the rolls run slightly faster than the other to give a shearing action, this action on the curled hulls tending to break the hour-glass cells within the hull and to provide apertures for good liquid absorption.

As illustrated in Figure 4.1b, the original hull has an outer layer or epidermis (22), consisting of palisade cells which are covered on top by a waxy cuticle, a middle layer consisting of hour-glass or I-cells (23), and an inner layer (24) facing the cotyledon in the seed. This inner layer comprises the spongy parenchyma consisting of several strata of flat boxlike cells and the aleurone cells which are filled with dense protein (aleurone). The exposed surface of the aleurone cells in the hull separated from the seed is also covered by a thin cellulose layer (25).

The plastic and curled hulls, under the action of the flaking rolls, while retaining their original cell organization, are nevertheless ruptured, the outer epidermis being ruptured and the hour-glass cells being broken apart or collapsed. The inner aleurone cell layer is also ruptured. The main change is in the collapsing of the hour-glass cells, and under the pressure of the flaking rolls, the palisade and aleurone layers move closer together to provide narrowed capillary passages. The narrowed passages are effective in the absorbing of liquid ingredients while at the same time the maintenance of the outer layers 22 and 24 protect and occlude these liquid ingredients within the flake, thus enabling the flake to have a relatively dry exterior and thereby to remain free flowing.

From the flaking rolls, the flakes may be drawn through the conduit 29 into the cyclone 30, and from thence, if desired, through a flake breaker 31 to reduce the size of the flakes, the broken product being received within the storage bin 32. From the storage bin 32, the flaked hulls may be passed through mixers 33 and liquids such as molasses or other nutritive material incorporated in the flakes.

Example: Soybean hulls were moistened by the addition of 5% water and sufficient live steam was introduced to bring the hull temperature to approximately 200°F. and the moisture content to approximately 19%. In about 5 minutes, the operation was completed, the shells coiling upon themselves to form shell rolls. These coiled hulls were then passed through flaking rolls to form flakes of approximately 0.005" thickness. The product was cooled by flashing off the moisture. The material thus obtained was silky, fluffy, soft, translucent, highly absorbent, and had lost all resemblance to the original material. The bulk density of this product was approximately 8 pounds per cubic foot. It was combined with approximately 30% molasses and the product air dried. The solid portions of the molasses appeared as particles upon the exterior surfaces of the flakes, (see Figure 4.1c), giving a dry product of low bulk density and possessing excellent handling characteristics.



### Pelleted Molasses-Containing Feed

A method for obtaining a hard noncrumbling pelleted feed containing molasses is described by N.F. Kruse (U.S. Patent 2,947,632; August 2, 1960; assigned to Central Soya Co., Inc.). Molasses is added to a meal carrier, the liquid molasses thoroughly penetrating the meal, and the resultant granular material is dried to a condition at which it flows readily. Steam is then introduced into the dried material containing molasses so as to distribute a thin layer of moisture on the surface of the particles to lubricate them, and the material is then fed through a pelleting machine.

Example: Soybean toasted meal was mixed with liquid molasses in the proportion of 12%, the molasses being sufficiently liquid to coat the particles of the meal. The meal was then passed through a steam tube dryer and the moisture reduced to approximately 10 to 12%. It is sufficient if the moisture be reduced to a point at which the material flows readily and can be readily handled in conveying and elevating equipment. The dried product was cooled, ground, and then subjected to steam, which gave a thin moisture coating to the molasses film. In addition to the molasses contained on the meal in the dry form, it was possible to add some liquid molasses on this feed product and yet maintain an effective pelleting operation.

The 12% molasses meal described above and in which the molasses coatings were dried was mixed with other meals and feed materials as described below, and this operation is compared with the operation in which the dried molasses mix was not employed and in which all of the molasses added was in the form of liquid molasses. Steam was added to the mixture containing the dried molasses coating prior to the pelleting operation, while no steam was added to the second operation in which all of the molasses was incorporated as liquid molasses.

### Low Protein Dairy Feed (Pelleted)

| Ingredient                                | Dried molasses-Meal Feed, Percent                          | Liquid Molasses Feed, Percent                                   |
|---|--|---|
| Ground Barley.....                        | 3.....   | 3.  |
| Cottouseed Meal.....                      | 2.....   | 2.  |
| Corn Gluten Feed.....                     | 14.....  | 14.   |
| Malt Sprouts.....                         | 5.....   | 5.  |
| Mineral Mix.....                          | 7.....   | 7.  |
| Ground Grain Screenings.....              | 31.....  | 30.   |
| 12% Molasses-Meal Mix Dried.....          | 25.....  | 0.  |
| Soybean Oil Meal.....                     | 0.....   | 22.   |
| Liquid Molasses.....                      | 13.....  | 17.   |
| Total Molasses (Reconstituted basis)..... | 17.....  | 17.   |
| Moisture of Mix.....                      | 10.8.....  | 13.5.   |
| Protein.....                              | 20.....  | 20.   |
| Soft Feed Mix Characteristics.....        | Relatively dry, not sticky and conveys without difficulty. | Wet and sticky. Hinders conveying. Will not bin satisfactorily. |
| Pelleting Rate.....                       | 23,200 lbs./hr.  | 10,000 lbs./hr.   |
| Pellet Quality.....                       | Hard, firm and smooth.                                     | Soft and crumble easily.  |

### Liquid Ruminant Supplement

R.I. Loomis and J.W. Algeo (U.S. Patent 3,248,224; April 26, 1966; assigned to Loomix, Inc.) have developed a process to provide a liquid ruminant supplement, readily varied, for controlling the ad libitum intake of protein, energy vitamin A, and phosphorus, as well

## Molasses and Flavorings

as drugs when necessary, and wherein a relatively unpalatable substance constitutes the primary ingredient vehicle rather than a more highly palatable substance, such as molasses.

A primary ingredient of the compositions, comprises condensed beet solubles, which is the principal carrier of the mixtures. By condensed beet solubles, is meant the residual product obtained by the partial removal of glutamic acid from the filtrate resulting from the Steffens process of recovering sugar from beet molasses; the Steffens process being well known in the art. The residual product of the Steffens process of recovering sugar from cane molasses can be used with equal effectiveness. Or, it is possible to employ the filtrate of the Steffens process without removal of the glutamic acid. Condensed beet solubles, unlike cane, corn or beet molasses, is an unpalatable liquid and by altering the level of the condensed beet solubles, the consumption of the supplement (which otherwise would fluctuate considerably in relation to the availability of dry range grasses or other forages) is controlled.

|                   |                         |          |
|-------------------|-------------------------|----------|
| <u>Example 1:</u> | Condensed beet soluble  | 65.000 % |
|                   | Molasses (beet or cane) | 28.976 % |
|                   | Urea solution (20% N)   | 6.000 %  |
|                   | Vitamin A (stabilized)  | 0.024 %  |

The 20% nitrogen urea solution comprises pure crystalline urea dissolved in water, and the vitamin A has been stabilized against oxidation for periods approximating 60 days by an aqueous mixture of antioxidants, such as the product sold commercially under the trademark "Lequivite". The formulation of Example 1 is efficacious as a liquid ruminant supplement where cattle or sheep are grazing in relatively mild or warm climates and native grasses are reasonably plentiful. This formulation affords approximately 23% of crude protein and supplies approximately 39% of new energy as well as approximately 5,000 international units of vitamin A per pound of supplement intake.

|                   |                                     |          |
|-------------------|-------------------------------------|----------|
| <u>Example 2:</u> | Condensed beet solubles             | 49.000 % |
|                   | Molasses (cane or beet)             | 21.976 % |
|                   | Urea solution (20% N)               | 2.000 %  |
|                   | Diammonium phosphate solution (50%) | 12.000 % |
|                   | Propylene glycol                    | 15.000 % |
|                   | Vitamin A (stabilized)              | 0.024 %  |

The amount of condensed beet solubles is appreciably reduced or adjusted in its relation to molasses, from the formulation of Example 1, and the chemical compound propylene glycol has been added. The formulation of Example 2 is a relatively fine liquid ruminant supplement for cattle grazing on rangelands of dry oat or wheat stubble and the like, or other dry forages, in appreciably cold regions. The propylene glycol present prevents the supplement from freezing and thereby provides a supplement which cattle will accept.

Propylene glycol has a two fold purpose, it serves as an antifreezing agent, and it readily converts by rumen microorganisms into propionic acid which is a primary energy source for ruminant animals. Hence, while the percentage of intake of crude protein in the formulation is about the same as in Example 1, the supply of net energy is close to 60% per pound of intake by reason of the additional energy supplied by the propylene glycol, which is highly advantageous for cattle feeding in extremely cold areas. The presence



of diammonium phosphate solution in this formulation also lends to its high effectiveness as a liquid ruminant supplement in cold region.

### Stable Feed Emulsions Containing Molasses

A process to produce a stable pumpable liquid emulsion, semiliquid emulsion or solid emulsion for animal feed purposes, in an inexpensive and efficient manner which emulsion includes molasses and livestock edible animal and vegetable fats and oils has been developed by M.D. Appleman (U.S. Patent 3,420,672; January 7, 1969; assigned to Jack J. Schroeder). The following formulations illustrate the precise ingredients in a variety of stable oil-molasses emulsions. All of the examples employ a fairly high loading of the fat or oil in molasses (30% by weight of fatty material by total weight of molasses and fatty material).

Example 1: The following ingredients are employed.

|  | <u>Parts by Weight</u> |
|--|------------------------|
| Water  | 50                     |
| Starch   | 0.3                    |
| Molasses — 70 Brix   | 350                    |
| Ammonium phosphate   | 20                     |
| Urea   | 40                     |
| Sodium benzoate  | 10.6                   |
| Sorbistat (potassium salt of sorbic acid)  | 0.6                    |
| Acidulated cottonseed oil  | 150                    |
| Tenox (various antioxidants such as butylated hydroxy anisole and butylated hydroxy toluene) | 0.01                   |

The starch was first added to the water and the water was then heated to boiling (100°C.). Upon heating of the water to between 50° to 70°C. a partial gel was produced. Upon heating further, the viscosity increased indicating further gelatinization of the starch. The boiled water-starch gel was then added to the molasses and stirred to uniformly distribute the starch. The water-soluble additives are then added to the molasses-starch system, i.e., ammonium phosphate, urea, sodium benzoate and the Sorbistat. The ammonium phosphate and urea are added for nutritional purposes; the benzoate and Sorbistat are preservatives. The cottonseed oil is then added along with the oil-soluble Tenox. The above mixture is emulsified by means of a high speed mixer and a stable emulsion resulted. The emulsion of Example 1 is a light gel but is somewhat pumpable at a temperature of 95°F.

Examples 2 to 8: The following Examples 2 to 8 are formulations showing the use of various types of fats and oils, as well as the use of varying amounts of starch. Examples 2 to 8 were formulated in the manner described in Example 1. The formulation of Example 2 is a stable, somewhat pumpable, emulsion at 95°F. The formulations of Examples 3 and 4 are stable and readily pumpable at 70°F. This is because of the use of only 0.03% starch in Examples 3 and 4 rather than 0.06% starch in Examples 1 and 2. By way of comparison, if the starch were omitted from any of the foregoing examples, the emulsions would break within a matter of several days.

## Molasses and Flavorings

| [Parts by weight]       |                  |                  |                  |                  |                  |                 |                  |
|-------------------------|------------------|------------------|------------------|------------------|------------------|-----------------|------------------|
|                         | 2                | 3                | 4                | 5                | 6                | 7               | 8                |
| Water.....              | 50               | 50               | 50               | 50               | 50               | 50              | 50               |
| Starch.....             | 0.3              | 0.15             | 0.15             | 0.15             | 0.15             | 0.15            | 0.15             |
| Molasses—70 Brix.....   | 350              | 350              | 350              | 350              | 350              | 350             | 350              |
| Ammonium Phosphate..... | 20               | 20               | 20               | 20               | 20               | 20              | 20               |
| Urea.....               | 40               | 40               | 40               | 40               | 40               | 40              | 40               |
| Kaoline.....            | 0.6              | 0.6              | 0.6              | 0.6              | 0.6              | 0.6             | 0.6              |
| Sorbistat.....          | 0.6              | 0.6              | 0.6              | 0.6              | 0.6              | 0.6             | 0.6              |
| Oil.....                | <sup>1</sup> 150 | <sup>2</sup> 150 | <sup>1</sup> 150 | <sup>3</sup> 150 | <sup>4</sup> 150 | <sup>5</sup> 50 | <sup>6</sup> 150 |
| Tenox.....              | 0.01             | 0.01             | 0.01             | 0.01             | 0.01             | 0.01            | 0.01             |

<sup>1</sup> Acidulated corn oil used.

<sup>2</sup> Acidulated cottonseed oil used.

<sup>3</sup> Fish oil.

<sup>4</sup> Tallow.

<sup>5</sup> Sewage fat.

<sup>6</sup> Olive oil.

**Examples 9 to 12:** Examples 9 and 10 below illustrate the preparation of a solid emulsion for the range feeding of cattle and are prepared in accordance with the directions given for Example 1, except that the starch is added directly to the molasses without any intermediate water-starch gel being made. Inert ingredients, such as bentonite or kaolin are added in order to still further increase the hardness of the solid emulsions of Examples 9 and 10 although it should be understood that such ingredients are not necessary to obtain the solid stable emulsion. Each of Examples 9 through 12 employs a different fat or oil.

Examples 11 and 12 further illustrate the preparation of a solid stable emulsion of molasses and oil, utilizing five times the amount of starch used in Examples 9 and 10, the total amount of starch being 6% by weight of the total weight of the molasses and oil. The formulations of these Examples 9 to 12 follow:

| [Parts by weight]       |                  |                  |                  |                  |
|-------------------------|------------------|------------------|------------------|------------------|
|                         | 9                | 10               | 11               | 12               |
| Molasses—70 Brix.....   | 350              | 350              | 350              | 250              |
| Ammonium Phosphate..... | 20               | 0                | 20               | 0                |
| Ammonium Sulfate.....   | 0                | 60               | 0                | 60               |
| Urea.....               | 20               | 20               | 40               | 0                |
| Starch.....             | 6                | 6                | 30               | 30               |
| Sodium benzoate.....    | 0.1              | 0.1              | 0.1              | 0.1              |
| Sorbistat.....          | 0.1              | 0.1              | 0.1              | 0.1              |
| Oil.....                | <sup>1</sup> 150 | <sup>2</sup> 150 | <sup>3</sup> 150 | <sup>4</sup> 150 |
| Bentonite.....          | 10               | 0                | 10               | 10               |
| Kaoline.....            | 0                | 10               | 0                | 0                |

<sup>1</sup> Sesame oil.

<sup>2</sup> Restaurant grease.

<sup>3</sup> Soybean oil.

<sup>4</sup> Acidulated soap stock.

## PREVENTION OF GEL FORMATION IN MOLASSES-PHOSPHORIC ACID SUPPLEMENTS

Various liquid feed supplements for animals, particularly ruminants, include phosphoric acid, or an equivalent soluble phosphate and a molasses. It has been found that the use of certain types of molasses result in gelation of the phosphoric acid-molasses mixtures to nonfluid masses at unpredictable periods.

### Use of Non-Phosphatic Acid

G.R. Weber and F.D. Miller (U.S. Patent 3,165,413; January 12, 1965; assigned to National Distillers and Chemical Corporation) have developed a process for the preparation of liquid animal feed supplements containing phosphoric acid and molasses which are



## Molasses and Flavorings

stable to gelling during storage wherein the molasses used normally has a high gelling tendency, by incorporating a small amount of a non-phosphatic acid into the liquid feed supplement.

To measure the relative tendencies of various molasses to gel and the effects on such tendencies produced by use of the hereindescribed process, a standard and realistic test for gelling was devised. An aqueous phosphatic gelling test solution with a pH of 2.7 was prepared containing 80.56 grams of monobasic sodium phosphate,  $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$ , and 11.90 g. of 85% orthophosphoric acid per liter of solution.

In the standard procedure, one part by weight of this test solution was blended uniformly with two parts by weight of the molasses, the blend placed in a stoppered cylinder or vial, and the vial placed in a water bath or incubator regulated to 108°F. (42°C.). The vial was examined periodically and the extent of gelling noted. Depending upon the molasses used, the start of gel formation was observed in as little as three minutes to as long as 90 days or more. The following table shows the test results recorded for a series of commercial samples of feed grade molasses:

| <u>Type</u>  | <u>Molasses</u>         |           | <u>Blend with Phosphatic<br/>Test Solution</u> |                     |
|--------------|-------------------------|-----------|--|---------------------|
|              | <u>Source of Sample</u> | <u>pH</u> | <u>pH</u>                                      | <u>Gelling Time</u> |
| Mexican cane | Pennsylvania            | 4.98      | 4.48   | < 2.0 hr.           |
| Mexican cane | Maryland                | 5.09      | 4.54   | < 1.5 hr.           |
| Mexican cane | Texas                   | 5.56      | 4.94   | < 1.5 hr.           |
| Blackstrap   | New York                | 5.40      | 4.69   | < 2.0 hr.           |
| Blackstrap   | Kentucky                | 5.35      | 4.74   | > 16 days           |
| Blackstrap   | Arizona                 | 5.05      | 4.50   | > 90 days           |

At a temperature of 108°F., gelling in the test blend within about two hours corresponds to gelling in a commercial sample made from the same molasses, stored at 108°F., within about one to three days; gelling in the test blend occurring between two and twenty-four hours corresponds to gelling in the commercial sample occurring in the three to six week range. Finally, where no gelling occurs within twenty-four hours in the test blend, the corresponding commercial sample resists gelling for over six weeks, these periods being of sufficient duration for the general use pattern of the product. It was found that it is not possible to predict the gelling tendencies of a molasses merely from its pH or to prevent such tendencies by adjusting the pH of mixtures of molasses and phosphoric acid to a particular range by varying the ratio of phosphoric acid to molasses.

Weber and Miller have found that generally, the use of up to about 12 parts by weight of concentrated sulfuric acid per 100 parts by weight of 75% phosphoric acid or its equivalent will prevent gelling in liquid feeds containing any particular feed grade molasses; preferably, from about 5 to 9 parts of concentrated sulfuric acid is used. This process is particularly effective in alleviating the gelling tendencies of a liquid feed supplement for ruminants containing about 1 to 12 parts by weight of ethanol per 10 parts by weight of urea per 1/2 to 5 parts by weight of phosphoric acid per about 70 to 175 parts by molasses, vitamin A,



and a number of soluble mineral salts. This liquid feed supplement is described in U.S. Patent 2,808,332.

The incorporation of about 0.025 to 0.45 parts by weight of sulfuric acid is effective for preventing gel formation here. Quantities of hydrochloric acid supplying the equivalent acidity as the sulfuric acid are generally effective, but less satisfactory from the standpoint of the usually less desirable chlorides introduced. The weaker non-phosphatic acids, e.g., acetic, tartaric, citric, and the like, are effective for use with molasses which exhibit moderate gelling tendencies, but are less preferred than sulfuric acid, particularly for use with the higher gelling molasses, due to the correspondingly greater quantities required to provide the equivalent acidity, and the relatively higher cost of such acids.

### Addition of Alkali Metal Polyphosphate

In a process developed by J.W. Lyons (U.S. Patent 3,325,289; June 13, 1967; assigned to Monsanto Company) it was found possible to prevent gelation of molasses-phosphoric acid feed supplements by adding a small amount of a water-soluble alkali metal or ammonium polyphosphate.

Sodium and potassium linear polyphosphates having a chain length of from about 3 to several hundred are preferred and particularly preferred compounds are sodium tripolyphosphate, sodium hexametaphosphate, Graham's salts and Kurrol's salts. In addition to their unexpected properties in preventing the gelation of mixtures of sugar-containing residues and ortho- and/or pyrophosphates, the water-soluble alkali metal polyphosphates are further advantageous in that they constitute a small but significant further source of phosphorous which is readily available to the animal when such compositions are incorporated in animal feed rations.

The amount of the water-soluble alkali metal polyphosphates which may be employed in the processes is an amount sufficient to maintain the compositions in a fluid state, and will generally depend upon the amount of ortho- and/or pyrophosphate in the compositions and also the degree of tendency of the sugar-containing residues to form a gel when mixed with the ortho- and/or pyrophosphate. Generally, from the about 0.1% to about 2.0%, preferably 0.1% to about 1.0% by weight of water-soluble alkali metal polyphosphate are used.

In a particularly preferred embodiment of this process, the compositions provided are fluid and comprise a mixture of (1) a major proportion of a cane or a beet molasses, (2) from about 2% to about 7% by weight of orthophosphoric acid, and (3) from about 0.1% to about 1.0% by weight of sodium tripolyphosphate, sodium hexametaphosphate and/or Graham's salt. In order to provide the compositions in a stable fluid state, it is desirable to mix the alkali metal polyphosphates with the other ingredients before gelation occurs. Thus, it has been found preferable to add the alkali metal phosphates to the sugar-containing residues prior to or simultaneously with the ortho- and/or pyrophosphates. By so-proceeding, it is possible to achieve compositions which are stable fluids and which will not gel on standing for periods of six months or longer.

Example: The following compositions having the proportions shown were prepared. The compositions were fluid and pourable after 23 weeks of storage in a laboratory cabinet at



## Molasses and Flavorings

| Ingredient                        | Composition Number |      |      |      |      |      |      |      |      |
|-----------------------------------|--------------------|------|------|------|------|------|------|------|------|
|                                   | 1                  | 2    | 3    | 4    | 5    | 6    | 7    | 8    | 9    |
| Cane Molasses.....                | 91.5               |      | 87.4 |      | 90.3 |      | 89.6 |      | 46.7 |
| Beet Molasses.....                |                    | 93.4 |      | 88.5 |      | 87.3 |      | 89.7 | 46.7 |
| Orthophosphoric Acid.....         | 6                  | 4    |      |      |      |      |      |      | 4    |
| Pyrophosphoric Acid.....          |                    | 1    |      |      |      |      |      |      | 1    |
| Di-ammonium Orthophosphate.....   |                    |      | 6    | 11   |      |      |      |      |      |
| Mono-ammonium Orthophosphate..... |                    |      | 6    |      | 9    |      |      |      |      |
| Trisodium Orthophosphate.....     |                    |      |      |      |      | 12   |      |      |      |
| Di-ammonium Pyrophosphate.....    |                    |      |      |      |      |      | 10   |      |      |
| Tetra-sodium Pyrophosphate.....   |                    |      |      |      |      |      |      | 10   |      |
| Sodium Tripolyphosphate.....      | 0.5                |      | 0.6  |      |      |      |      | 0.7  | 0.3  |
| Sodium Hexametaphosphate.....     |                    | 0.6  |      | 0.5  |      |      | 0.4  |      | 0.3  |
| Graham's Salt.....                |                    |      |      |      | 0.7  |      |      |      |      |
| Kurrol's Salt.....                |                    |      |      |      |      | 0.7  |      |      |      |
| Water.....                        | 2                  | 1    |      |      |      |      |      |      | 1    |

100°F. On the other hand, compositions similar to compositions 1 through 9, but which differed in that they did not contain alkali metal polyphosphates formed thick nonpourable gels within 12 hours.

### AMMONIATED SUGARS

#### Ammoniated Low Grade Sugar Products

W.R. Fetzer (U.S. Patent 3,020,157; February 6, 1962; assigned to Standard Brands, Inc.) has developed a process to provide a feed supplement for ruminants by utilizing a sugar-containing material normally having a high reducing sugar content but a low protein content and ammoniating it in such a manner as to substantially increase its protein-equivalent nitrogen content without needless loss of the reducing sugars.

The process involves the addition of the ammonia in the proportion of between 1.0% and 5.5%, based on the dry substance weight of the sugars present in the syrup being treated. Acidic material is added so that the pH is maintained within a range of 4.0 to 7.5 pH. An ammonium salt may be used to supply the acidulating agent and part of the ammonia, or if desired, a free acid could be used in addition to or without the ammonia salt, and all of the ammonia employed, or most of it, could be added as ammonia.

The process is preferably performed in a closed reaction vessel or kettle, primarily to prevent the loss of ammonia gas. Such a vessel may be a jacketed kettle six feet in diameter and approximately twenty feet in depth, equipped with an agitator. The ammonia system consists of an ammonia cylinder on a dial type weighing scale, connected through a flexible hose to an iron pipe system leading to the reaction kettle. A shut off valve is a part of the cylinder assembly; and another shut off valve is at the kettle. The pipe system in the kettle consists of a pipe leading to a circular ring in the bottom of the kettle which is provided with a large number of small holes. There is an additional small hole 1/64 of an inch in diameter in the top of the pipe below the inner surface of the kettle and above the liquid level. The acid or ammonium salt is added gradually in any appropriate manner which will avoid localized over-acidulation.

In performing the process any one of various sugar-containing syrups may be used, but for

purposes of economy a low-grade sugar product such as corn sugar molasses (hydrol), citrus molasses, cane or beet molasses, wood sugars and liquors containing wood sugar or the like are used. The ratio between the ammonia and the acidic material required to maintain the required pH range may be affected by the buffer power of the syrup, and therefore, alterations must be made where necessary to compensate for changes in the buffering capacity of the syrup being treated.

In operation, the sugar-bearing material is placed in the reaction kettle described above, and heated by any convenient means, such as steam on the jacket, preferably to a temperature within the range of 140° to 220°F. Starting materials in the density range of 39° to 45° Bé., or 73° to 85° Brix, are preferred for convenience in pumping, stirring and general economy, and materials at such densities are conveniently treated in this process in the range of 140° to 220°F., although any temperature from ambient to 220°F. may be employed. If desired, part or all of the chemical reagents may be added to the cold sugar bearing material before this is heated, but it is more convenient to add at least that part of the ammonia required in excess of the stabilizing salts after the mass has been warmed.

The desired quantity of liquid ammonia is fed into the pipe and the major part thereof evolves as gas through the ammonia distributing ring and some through the small orifice at the top of the pipe system. The liquid ammonia in changing from a liquid to a gas within the system obtains its latent heat of vaporization from the syrup, which offsets some of the temperature increase resulting from the heat of reaction. When the desired weight of ammonia has been added, the cylinder valve is closed, the liquid ammonia in the pipe system allowed to complete its evaporation as gas in the kettle, and the valve adjacent the kettle is closed. The small orifice in the top of the pipe system prevents a flashback of sugar produced by the rapid absorption of the ammonia by the sugar syrup.

After the chemicals have been added the mixture is held at the reaction temperature until the desired reactions have been obtained. Thereafter the product needs only to be cooled and will remain stable. The final product realized is stable under normal room temperature conditions with respect to ammonia release over the pH range 4.0 to 7.5 and is essentially free from untoward decomposition products.

Example 1: Corn sugar hydrol was ammoniated at 200° to 220°F. as follows.

|   |             |
|---|-------------|
| Hydrol, as defined by analysis<br>on the following page | 1000 pounds |
| Hydrochloric acid (calculated<br>as anhydrous HCl)      | 24 pounds   |
| Anhydrous ammonia, NH <sub>3</sub>                      | 29 pounds   |
| Time for addition of chemicals                          | 30 minutes  |
| Time of heating after chemical<br>additions             | 30 minutes  |

Cool to below 100°F.



## Molasses and Flavorings

|  | Analyses            |                    |
|--|---------------------|--------------------|
|  | Before<br>Treatment | After<br>Treatment |
| Dry substance, percent   | 73.5                | 69.8               |
| Reducing sugars, dry basis (D.B.), %                               | 73.4                | 59.0               |
| Total crude protein equivalent,<br>D.B., percent                   | 0.3                 | 24.6               |
| Uncombined NH <sub>3</sub> calculated as<br>protein, D.B., percent | —                   | 10.3 <sup>1</sup>  |
| pH   | 4.4                 | 7.3                |
| Color  | 2.0                 | 3.0                |

<sup>1</sup> 1.8 as NH<sub>3</sub>

Example 2: Citrus molasses was ammoniated at 200° to 220°F. as follows.

|  |              |
|--|--------------|
| Citrus molasses  | 1,000 pounds |
| Ammonium sulfate (containing 14.5<br>pounds of NH <sub>3</sub> ) | 56.2 pounds  |
| Anhydrous ammonia  | 14.5 pounds  |
| Time for addition of chemicals                                   | 30 minutes   |
| Time of heating after chemical additions                         | 30 minutes   |

Cool to below 100°F.

|  | Analyses            |                    |
|--|---------------------|--------------------|
|  | Before<br>Treatment | After<br>Treatment |
| Dry substance, percent   | 72.2                | 69.6               |
| Reducing sugars, dry basis, percent                                | 27.7                | 15.8               |
| Sucrose, dry basis, percent  | 30.2                | 27.6               |
| Total crude protein equivalent, D.B.,<br>percent                   | 7.1                 | 27.1               |
| Uncombined NH <sub>3</sub> calculated as<br>protein, D.B., percent | —                   | 11.8               |
| pH   | 4.4                 | 5.5                |
| Color  | 0.2                 | 2.0                |

### Ammoniated Sweetened Feed

In another process (U.S. Patent 3,020,158; February 6, 1962; assigned to Standard Brands Inc.) W.R. Fetzter utilizes a somewhat similarly ammoniated syrup in a feed composition. However, instead of continuing the heating cycle until the ammonia has fully reacted with the sugars, the cycle is interrupted and a dry solid feedstuff ingredient is admixed with the

partially reacted syrup, after which the mixture is dried with heat to the final desired moisture content. The partially reacted ammonia-syrup mixture may be cooled below the reaction temperature before or during the mixing with the solid ingredients, and the mixture thereafter reheated to complete the reaction and to dry the mixture to a desired final moisture content. The following examples illustrate the process.

Step A — Typical Conditions for Partial Ammoniation of Hydrol: One thousand pounds of hydrol at 43.3° Bé. of 73.5% dry substance, contains 735 pounds of dry substance. For a 5% level of ammoniation, 36.75 pounds of total ammonia ( $\text{NH}_3$ ) is required. Ammonium chloride was selected as the acidic constituent to control the pH. For suitable control of the pH in these examples, 52.1 pounds of ammonium chloride was employed, which contributed 45% of the total ammonia required, i.e., 16.55 pounds of  $\text{NH}_3$ . The balance of the ammonia, 55% of the total, or 20.2 pounds, was added as anhydrous ammonia. The reaction mixtures were heated at 200°F. for various lengths of time.

Step B — Typical Conditions for Mixing Partially Ammoniated Hydrol into a Solid Feed: The liquid products of these several experiments were subsequently mixed with solid feed ingredients to produce an ammoniated sweetened feed. In each of these cases, the following ingredients were combined:

- 7.5 pounds Corn Gluten Meal at 10% moisture, containing 41% crude protein.
- 33.7 pounds Corn Oil Meal containing 10% moisture and 21% crude protein.
- 58.8 pounds partially ammoniated hydrol, containing about 30% moisture, and about 26% crude protein equivalent.

These wet mixtures were then dried in the customary manner for feedstuffs; namely, to 5% and to 1% residual moisture. The analyses of the partially ammoniated hydrols and their respective dried feed products containing them are shown in the tables on the following page. Terms used in these tables are defined as follows:

Crude protein equivalent —  $\text{N} \times 6.25$ .

"Free"  $\text{NH}_3$  — Determined as described in "Technical Methods of Analysis"

by R.C. Griffin, McGraw-Hill Book Co., Inc., Second Edition, 1927, p. 91.

pH of syrups — Glass electrode value obtained on the syrup at full concentration.

pH of feeds — Glass electrode value of an extract of 10 g. of feed diluted to 100 ml. with distilled water.

Color — The Lovibond value, caramel series 52, in a one-inch cell employing a solution obtained from 1 g. of material diluted to 1,000 ml. with distilled water.

D.E. — Reducing sugars calculated as dextrose and expressed as a percentage of the total dry-substance.

When the mixture of ammoniated molasses and solid feedstuff is being dried in such a conventional commercial feed dryer as a steam heated "Louisville" feed dryer the mixture will dry down to between 2 and 5% moisture content in about one hour, and will not on the average be heated above 212°F., more generally around 200°F. In such time and at such temperature the ammonia reaction does not proceed too far and the product will have a



## Molasses and Flavorings

### Analyses of Control Hydrol and of Partially Ammoniated Hydrols and of Sweetened Feeds Containing Them

|   | Control Hydrol | Liquid Ammoniated Hydrols       |       |       |       |       |
|---|----------------|---------------------------------|-------|-------|-------|-------|
|   |                | Time Held at 200° F. in minutes |       |       |       |       |
|   |                | 37                              | 60    | 120   | 180   | 240   |
| Ammonia Added, Percent of D.S.                        | 0.0            | 5.0                             | 5.0   | 5.0   | 5.0   | 5.0   |
| Dry substance, percent                                | 73.6           | 71.81                           | 71.64 | 70.46 | 69.92 | 68.62 |
| D.E.  | 73.2           | 55.9                            | 55.2  | 51.8  | 46.5  | 45.0  |
| "Crude Protein Equivalent" D.B.                       | 0.2            | 25.5                            | 25.8  | 26.1  | 25.5  | 25.8  |
| "Free" Ammonia D.B.                                   | 0.0            | 2.9                             | 2.8   | 2.4   | 2.1   | 1.9   |
| "Crude Protein Equivalent" of the "Free" Ammonia D.B. | 0.0            | 14.04                           | 14.92 | 12.36 | 10.82 | 9.79  |
| pH  | 4.2            | 7.4                             | 7.1   | 6.6   | 6.1   | 5.3   |
| Color   | 0.2            | 1.5                             | 2.0   | 3.8   | 6.2   | 7.0   |

#### Sweetened Feeds Made with Above Hydrols and Dried to 5% Moisture Content

|   |      |      |      |  |  |      |
|---|------|------|------|--|--|------|
| Moisture  | 4.8  | 5.1  | 5.9  |  |  | 4.3  |
| D.E.  | 38.4 | 30.4 | 28.9 |  |  | 21.7 |
| "Crude Protein Equivalent" D.B.                   | 13.1 | 26.8 | 26.8 |  |  | 26.8 |
| "Free" Ammonia D.B.                               | 0.05 | 1.10 | 1.12 |  |  | 0.76 |
| "Crude Protein Equivalent" of "Free" Ammonia D.B. | 0.26 | 5.67 | 5.70 |  |  | 2.91 |
| pH  | 4.3  | 6.8  | 6.8  |  |  | 4.6  |
| Color   | 0.1  | 1.2  | 1.5  |  |  | 4.3  |

#### Sweetened Feeds Made with Above Hydrols and Dried to 1% Moisture Content

|   |      |      |      |  |  |      |
|---|------|------|------|--|--|------|
| Moisture  | 1.3  | 1.5  | 1.1  |  |  | 0.79 |
| D.E.  | 36.7 | 24.7 | 21.8 |  |  | 18.9 |
| "Crude Protein Equivalent" D.B.                   | 13.0 | 26.8 | 26.8 |  |  | 26.8 |
| "Free" Ammonia D.B.                               | 0.05 | 0.45 | 0.29 |  |  | 0.25 |
| "Crude Protein Equivalent" of "Free" Ammonia D.B. | 0.26 | 2.32 | 1.49 |  |  | 1.29 |
| pH  | 4.4  | 4.5  | 4.2  |  |  | 3.8  |
| Color   | 0.2  | 5.3  | 6.3  |  |  | 7.3  |

negligible tendency to stick or compact when subsequently cooled to ambient temperatures. Thus even when dried only to 5% moisture it has been found to be satisfactory, whereas the prior known sweetened feeds usually require drying to less than 2.5%. Drying of the product to as low as 1% is not recommended as being unnecessary and because of the adverse effect on color and final D.E.

## FLAVORINGS

It is common practice to mix flavoring materials with animal feeds to make such feeds more tempting and palatable.

### Antioxidant Flavoring

During storage the fatty nutrients of the feed undergo oxidative deterioration which results in rancidity, off-odors and off-tastes in the feed thereby making it unpalatable to the livestock. To overcome this problem artificial flavoring has been added to livestock feed to

mask the off-flavor and off-odor. The flavorings too are subject to oxidative deterioration, and experience a change in flavor and a diminished flavoring effect.

T.B. Tribble (U.S. Patent 3,051,572; August 28, 1962) has developed a process to provide an antioxidant flavoring composition which, when applied to a fresh feed, will preserve its fresh flavor and odor with a greatly reduced quantity of flavoring. This method consists of homogeneously mixing fresh livestock feed, the fatty nutrient content of which has not undergone substantial oxidation, with from about 0.25% by weight to about 0.04% by weight of the antioxidant flavoring. The antioxidant flavoring may be homogeneously mixed with the feed by any suitable method such as spraying or fogging liquid antioxidant flavoring onto the feed or dusting or otherwise mixing powder-form antioxidant flavoring into the feed.

This process is not limited to any particular flavorings, but involves the use of substantially all livestock feed flavorings. Many food-grade antioxidants may be employed. Commercial compositions having particularly suitable properties are available such as Tenox II, containing about 20% by weight of butylated hydroxyanisole, about 6% by weight of propyl gallate, about 4% by weight of citric acid and about 70% by weight of propylene glycol.

|   |           |               |
|---|-----------|---------------|
| <u>Example — Antioxidant Flavoring:</u> | Tenox II  | 15.6 % weight |
|   | Anise oil | 84.4 % weight |

A feed preparation containing 0.05% by weight of this antioxidant flavoring, 11% animal grease and 88.95% feed was composited and found to have flavorstatic properties. The feed preparation of antioxidant flavoring was compared with three other feed preparations. These consisted of the same feed without any additives at all, the feed with 0.05% by weight of anise oil, and the feed with 0.05% by weight of Tenox II.

All four feed preparations were stored under identical conditions for three weeks during which the temperature varied from a low of 68°F. to a high of 90°F. and then were examined and tested. The feed preparation consisting only of feed and animal grease had developed strong rancid off-odors which were highly objectionable. The feed preparation consisting only of feed, animal grease and anise oil when freshly prepared had the delicate aroma of anise oil, but after the three week storage period all anise aroma was gone and a rancid off-odor indistinguishable from the unflavored feed preparation prevailed.

The feed preparation consisting of feed, animal grease and Tenox II had no rancid smell and the feed preparation prepared with the above antioxidant flavoring had no rancid smell, but instead it still had the delicate aroma of anise oil, although it was not as strong as when first composited. Following the three week storage period, the four feed preparations were tested as to animal acceptability in free-choice animal feeding tests. In all tests the feed preparation prepared with antioxidant flavoring given above was the only feed preparation that any animal would eat.

### Flavored Non-Carbohydrate Sweetener

Another process of T.B. Tribble (U.S. Patent 2,932,571; April 12, 1960) is preparation of a flavored sweetener which makes use of a non-carbohydrate sweetener. He discovered that a synergistic action occurs between the livestock feed flavorings and the concentrated



## Molasses and Flavorings

sweeteners when they are combined which results in an intensification of the flavor and sweetness of such a flavoring and sweetener in the composition and the masking, if not indeed the elimination, of the objectionable characteristic taste of the concentrated sweetener. The following formulas are illustrative of the additives; all percents are by weight.

### Formula No. 1 (dry powder)

|                  |        |
|------------------|--------|
| Anise oil        | 0.1 %  |
| Saccharin sodium | 1.0 %  |
| Corn sugar       | 98.9 % |

### Formula No. 2 (dry powder)

|                  |        |
|------------------|--------|
| Anise oil        | 1.0 %  |
| Saccharin sodium | 10.0 % |
| Corn sugar       | 89.0 % |

### Formula No. 3 (dry powder)

|                  |      |
|------------------|------|
| Anise oil        | 10 % |
| Saccharin sodium | 90 % |

### Formula No. 4 (liquid emulsion)

|                  |        |
|------------------|--------|
| Anise oil        | 2.0 %  |
| Saccharin sodium | 20.0 % |
| Vegetable gum    | 1.0 %  |
| Water            | 77.0 % |

### Formula No. 5

|                          |      |
|--------------------------|------|
| Saccharin sodium         | 1 %  |
| Molasses (dry or liquid) | 99 % |

### Formula No. 6 (dry powder)

|                  |         |
|------------------|---------|
| Anise oil        | 0.05 %  |
| "Sucaryl" sodium | 50.00 % |
| Corn sugar       | 49.95 % |

### Formula No. 7 (dry powder)

|                  |        |
|------------------|--------|
| Anise oil        | 0.1 %  |
| "Sucaryl" sodium | 99.9 % |

As a replacement for cane sugar in livestock feeds, 10 pounds of Formula No. 1, 1 pound of Formula No. 2, 0.1 pound of Formula No. 3, 0.5 pound of Formula No. 4, 10 pounds of Formula No. 5, 3.3 pounds of Formula No. 6, and 1.7 pounds of Formula No. 7,

respectively, have proved to be about the equivalent of 100 pounds of cane sugar. 0.5 to 2 lb. of flavored sweetener is used per ton of livestock feed.

To test the taste appeal of these compositions, baby pigs were chosen because they are considered to be difficult livestock to wean over to commercial starter feeds. Feed sweetened with cane sugar according to the prior art and feed treated with the flavored sweetener were placed in each of a plurality of pens and the baby pigs in each pen were given a free choice of the thus sweetened and treated feeds over the same period of time. The pigs in each pen consumed more than twice as much of the feed treated with the flavored sweetener of this process than of the sugar sweetened feed.

An advantage of the flavored sweetener is to intensify the flavor and sweetness of such natural nutritionally active feed additives as molasses, carob, and honey, and extend the use of such other carbohydrate feed ingredients having flavor values but which cannot be used in excessive quantities because of the ill effects they may have on the dietary system.

### Concentrated Flavoring Premix

A process for preparing a premix containing high concentrations of flavoring has been developed by P.Q. Card and N.K. Stanton (U.S. Patent 2,921,853; January 19, 1960; assigned to The Northern Trust Company). This is achieved by using the hardest part of the corn cobs (the hard woody ring) as carrier. In preparing a premix employing hard woody ring as a carrier, it is the dust separation of the corn cob grinding process that is used which was formerly considered to be waste. The hard woody ring is added in a suitable quantity to a mixer which may be a horizontal ribbon mixer having a sifter at one end thereof.

While the precise quantity of hard woody ring placed in the mixer is not critical, quantities in the order of about 200 pounds will comprise a convenient batch size. When added to the mixer, the hard woody ring preferably has a moisture content of less than 6% and it constitutes a mixture of two or more different particle sizes, ranging from 50 to 80 mesh but preferably 50 and 80 mesh. These two different particle sizes are employed, for in admixture they give a reddish or brown cast to the product after mixing which is commercially advantageous and thereafter there is very little color change with ageing. Further, however, the 50 and 80 mesh particles afford a more compact end product in that the smaller particles interlace the larger ones. Next a desiccant is mixed with the readied woody ring to further lower the moisture content thereof, and tricalciumphosphate has been found to be satisfactory.

The mixer is next energized, and the flavoring oil or oils are added slowly to the agitated woody ring and are therefore preliminarily mixed therewith. The flavoring oils are added in sufficient quantity so as to provide the desired proportion thereof to the woody ring. While the woody ring will absorb up to from 45 to 50% by weight of flavoring oils, it is preferable to mix in a somewhat smaller proportion of flavoring as, for example, from 25 to 35%. The reason for this is that such a smaller percentage affords a considerable safety margin in that if the premix product is exposed to extremely high temperatures (as, for example, up to and above 180°F.), there still will be no danger of the liquid flavoring separating from the woody ring carrier.

One or more liquid flavoring materials may be added to the woody ring. These include



essential oils, terpeneless oils, infused oils, oleoresins, solid extracts, aromatic chemicals, balsams, powdered extracts, animal derivatives, powdered dry spices, perfume oils, fixed oils, solvents, emulsifying agents, fractionated vegetable agents and tinctures. A mixture of various oils is taken from these and in this case the oils are first mixed together in a mixer. A proportion of about five to seven and one-half gallons of oil to a 200 pound batch of woody ring will afford the desired 25% of liquid flavoring to the woody ring, and such a quantity of flavoring liquid may be slowly added to the mixer in a period of about five minutes.

Mixing is continued at room temperature — that is, from about 50° to 85°F. — for a period of about 15 to 20 minutes. It has been found that the mixing time will vary with the viscosity of the liquid flavoring, and with a higher viscosity a greater mixing time is necessary. An elevation in temperature occurs during the mixing which must be considered in establishing the mixing time. Further, the mixing appears to have the effect of tearing the liquid apart to get it dispersed into the woody ring.

After the mixing has been carried to completion, the premix is fed into a flexible bag or container such as a polyethylene bag, which is essentially moisture-resistant and therefore will have the effect of protecting the premix from rain. There is little need, however, to protect the premix from simple high humidity because it exhibits substantially no tendency to absorb moisture. It may also be noted that any residual moisture in the woody ring during the mixing thereof may be displaced by the oil flavoring for it has the tendency to drive off moisture from the woody ring carrier.

The bag is placed within a container which may be a fiber drum, and thereafter a press or plunger is pushed downwardly against the filled bag to compress it into the smaller container. Ordinarily, the premix material is compressed by about 20% of its volume to force it into the smaller container. This has the advantage of requiring less space for storage and shipment, etc., and it does not have the effect of caking the premix.

## ESTROGENS AS GROWTH STIMULATORS

It has been known for a long time that trace amounts of estrogens have growth promoting properties for domestic animals. Diethylstilbestrol is commonly administered for this purpose in the raising of beef cattle and sheep. The processes covered in this section deal with the administration, handling and stability of such diethylstilbestrol preparations. Included are other newer compounds with estrogenic activity, which likewise exhibit growth stimulating properties in animals.

### COMPOUNDS FROM CULTURES OF GIBBERELLA

#### Gibberellin

Nutritionally balanced animal feed compositions containing gibberellin have growth promoting properties. (J.R. De Zeeuw, G.A. Donovan and W.C. Sherman; U.S. Patent 2,943,938; July 5, 1960; assigned to Chas. Pfizer & Co., Inc.) In general, gibberellin may be used in animal feeds at a concentration level of from about 0.0001 mg. to about 10 mg. per kg. in order to obtain the unusually high degree of response in growth promotion. The preferred proportion is a concentration level in the range of from about 0.01 mg. to 1.0 mg. of gibberellin per kg. of animal feed.

Gibberellin is a plant-growth promoter which is produced by fermenting a nutrient medium with Gibberella fujikuroi. Gibberellin is defined as a mixture comprising three different and distinct chemical compounds, viz., gibberellin A<sub>1</sub>, gibberellin A<sub>2</sub>, and gibberellin A<sub>3</sub> (gibberellin X or gibberellic acid), although the term gibberellin may also be applied to any one of the individual components.

The gibberellin used was prepared in accordance with the procedure described by Stodola et al in the Archives of Biochemistry and Biophysics, vol. 54, pp. 240-245 (1955), and it was isolated by extracting at room temperature the resulting fermentation broth with methyl ethyl ketone using a Podbielniak centrifugal extractor, concentrating the extract under reduced pressure to precipitate non-gibberellin materials, and further reducing the extract to precipitate pure crystalline gibberellins. The gibberellin so obtained is actually a multi-component mixture comprising the following individual components, based on counter-current distribution as well as on paper and column chromatography studies: 70% gibberellic acid (gibberellin X or gibberellin A<sub>3</sub>) and about 20% gibberellin A (gibberellin A<sub>1</sub> and A<sub>2</sub>); the remaining 5 to 10% consists of less active materials.



## Estrogens as Growth Stimulators

**Example 1:** The growth experiments with gibberellin were conducted on Nichol's White-Cross Chicks kept in electrically heated brooders on raised wire floors. The day old chicks were divided into lots of five males and five females per compartment, replicated four times per treatment. The basal diet employed was nutritionally balanced. The birds were individually weighed and records of feed consumption by lot were maintained. The results obtained are presented in the following table.

| Supplement                      | 28-day Weight (g.) | Growth Index | Feed Efficiency |
|---------------------------------|--------------------|--------------|-----------------|
| Control.....                    | 494                | 100          | 146             |
| Gibberellin, 0.0001 mg./kg..... | 495                | 100.2        | 143             |
| Gibberellin, 0.001 mg./kg.....  | 524                | 106.1        | 144             |
| Gibberellin, 0.01 mg./kg.....   | 501                | 101.4        | 146             |
| Gibberellin, 0.1 mg./kg.....    | 519                | 105.1        | 143             |
| Gibberellin, 1.0 mg./kg.....    | 505                | 102.2        | 146             |
| Gibberellin, 10.0 mg./kg.....   | 516                | 104.5        | 144             |
| Penicillin, 5.5 mg./kg.....     | 502                | 101.6        | 144             |

**Example 2:** Wether lambs were treated with nutritionally balanced animal feed compositions containing gibberellin at the level of one gram per ton. The lambs were divided into groups of twelve and each group was replicated twice per treatment.

| Supplement                      | 28-day Weight (g.) | Growth Index | Feed Efficiency |
|---------------------------------|--------------------|--------------|-----------------|
| Control.....                    | 450                | 100          | 1.59            |
| Gibberellin, 0.0001 mg./kg..... | 456                | 101.3        | 1.57            |
| Gibberellin, 0.001 mg./kg.....  | 484                | 103.1        | 1.57            |
| Gibberellin, 0.01 mg./kg.....   | 471                | 104.6        | 1.60            |
| Gibberellin, 0.1 mg./kg.....    | 461                | 102.2        | 1.60            |
| Gibberellin, 1.0 mg./kg.....    | 459                | 102.2        | 1.58            |

### Estrogenic Substance from *Gibberella Zeae* Culture

F.N. Andrews and M. Stob (U.S. Patent 3,196,019; July 20, 1965; assigned to Purdue Research Foundation) found that an anabolic composition which produces weight gain in meat-producing animals is readily produced by cultivating the organism *Gibberella zeae* (Gordon) on a suitable nutrient medium. The anabolic substance is prepared in the following way.

**Example 1 — Inoculum:** A spore sand culture containing *Gibberella zeae* (Gordon) NRRL-2830 was aseptically placed in a sterile tube containing 15 ml. of Czapek's-Dox solution and a small amount of agar. This medium was then incubated for about 168 hours at approximately 25°C. At the end of the incubation period, the medium was washed with 5 ml. of sterile deionized water and transferred to a sterile tube containing 45 ml. of Czapek's-Dox solution. The contents of the tube were then incubated for about 96 hours at about 25°C. after which the material was available for use in inoculation of a fermentation medium.

**Example 2 — Fermentation:** To a 2 liter flask were added 300 grams of finely divided corn. The flask and its contents were then sterilized and after sterilization 150 ml. of sterile deionized water were added. To the mixture in the flask were then added 45 ml. of the inoculum prepared by the process of Example 1 and the material was thoroughly mixed. The mixed material was then incubated for about 20 days at 25°C. in a dark room in a water-saturated atmosphere.

**Example 3 — Recovery of Anabolic Substance:** A 300 gram portion of fermented material produced by the method of Example 2 was placed in 500 ml. of deionized water and slurried. The slurry was then heated for about 15 minutes at 75°C., 300 grams of filter aid were then added and the material was filtered. The solid filtered material containing the anabolic substance was then air dried, and 333 grams of the dried cake were then extracted with 500 ml. of ethanol. This procedure was repeated three more times. The ethanol extract was then dried under vacuum to give 6.84 grams of solid material. This solid material was then dissolved in 20 ml. of chloroform and extracted with 30 ml. of an aqueous solution containing 5% by weight of sodium carbonate having an adjusted pH of about 11.2. The extraction process was repeated seven more times.

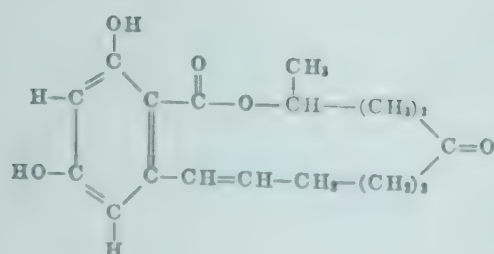
The pH of the sodium-carbonate extract was then adjusted to 6.2 with hydrochloric acid, to yield an anabolic substance-containing precipitate. The precipitate and the aqueous sodium carbonate extract were then each in turn extracted with 75 ml. of ethyl ether. This procedure was repeated three more times to yield a light yellow ethereal solution, which was then dried to yield 116 mg. of solid anabolic substance. This material was then subjected to multiple transfer countercurrent distribution using 100 tubes and a solvent system consisting of two parts chloroform and two parts carbon tetrachloride as the lower phase and four parts methanol and one part water as the upper phase, all parts by volume. The solid material obtained from the multiple transfer countercurrent distribution was then tested for physiological activity according to the well known mouse-uterine test.

As conducted, the mouse-uterine test consisted of feeding solid material produced from drying 5 ml. of the upper phase and 5 ml. of the lower phase from each of the selected tubes in a standard 75 gram feed to five mice for a five day period during which period all of the feed was consumed. At the end of the period, the animals were weighed and the uteri were removed and weighed. A positive response to the test was produced with the most physiologically active material being contained in tubes 30 through 40. The solid material collected from tubes 30 through 40 weighed 59 mg.

Ultraviolet absorption studies have been conducted on a methanolic solution of the estrogen. The curve shows an absorption maximum at 236, 274, and 313 millimicrons and an absorption minimum at 255 and 300 millimicrons. The anabolic substance shows a melting point of 164° to 165°C. The anabolic substance shows the following optical rotation in methanol:

$$[\alpha]_D^{25^\circ} = -109.5^\circ.$$

It has the structural formula:



The new anabolic substance is an effective growth promoting material for use in animal nutrition. The results of lamb growth studies utilizing eight lambs fed a commercial type ration

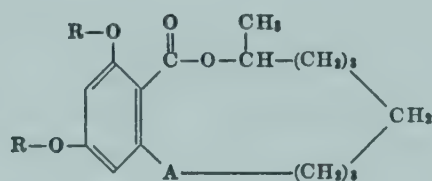


for 28 days showed that the average daily weight gain of these lambs was 0.27 lb. The average daily weight gain of lambs treated with 33 units of anabolic substance was 0.38 lb. which represents a 40% improvement in weight gain. One unit of anabolic substance is the amount of that substance needed to produce the response given by 1 microgram of diethylstilbestrol.

### Estrogenic Compounds from Fermentation Estrogenic Substance

These four processes developed by E.B. Hodge, P.H. Hidy and H.L. Wehrmeister, all assigned to Commercial Solvents Corporation, are for the preparation of estrogenic compounds with growth promoting properties. The compounds can be blended with ordinary feed containing nutritional values in an amount sufficient to produce the desired rate of growth and can be thus fed directly to the animals, or the compounds can be suspended in a suitable injection suspension medium such as peanut oil and injected parenterally. These compounds can be produced from the anabolic estrogenic substance described previously by Andrews and Stob (U.S. Patent 3,196,019). The solid material obtained by countercurrent distribution in that process is here referred to as fermentation estrogenic substance (FES).

The compounds described in U.S. Patent 3,239,341; March 8, 1966, have the formula:

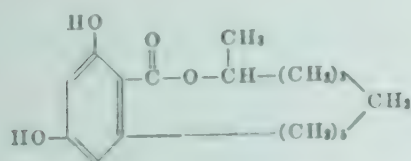


where A is selected from the group consisting of  $-\text{CH}=\text{CH}-$  and  $-\text{CH}_2\text{CH}_2-$ , and R is selected from the group consisting of hydrogen, lower alkyl and lower saturated acyclic acyl.

**Example 1 — Dihydro FES:** Two 10 gram portions of FES, each in 200 milliliters acetic acid, were catalytically reduced at room temperature in the presence of 1.2 grams of PdO catalyst at a hydrogen pressure of about 45 psi. The combined reduction mixtures were heated to boiling, filtered, and the filter cake was washed with 50 milliliters of hot acetic acid. The cooled filtrate was added, with stirring, to 2 liters of water. The mixture was stirred for 15 minutes and the white solid was collected by filtration, washed and dried in a vacuum desiccator to yield 19.1 grams of dihydro FES having a melting point of  $191^\circ$  to  $193^\circ\text{C}$ .

The dihydro FES (1 gram) is added slowly with cooling (ice-bath), to a mixture of 5 cc of ethylene dithiol, 0.25 gram of freshly fused zinc chloride and 2 grams of anhydrous sodium sulfate, contained in a microflask. The mixture is maintained at  $5^\circ\text{C}$ . for 20 hours and then at room temperature for 4 hours, whereupon it is poured into 50 cc of ice and the precipitate is collected and subjected to hydrogenolysis. To the reaction product is added 100 cc of 90% ethanol and 15 grams of Raney nickel catalyst and the mixture is refluxed until the reaction is complete. The nickel is removed by centrifugation and is washed several times with hot ethanol by centrifugation followed by decantation, and the centrifugates are combined. The mixture is evaporated to dryness and the residue is suitably recrystallized to yield a compound having the formula shown on the following page.

## Estrogens as Growth Stimulators

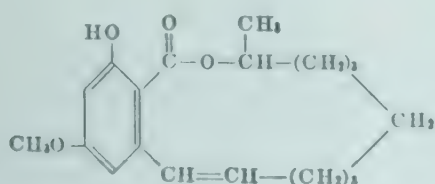


**Example 2 — Monomethyl FES:** Nitrosomethylurea in an amount of 1.2 grams was slowly added to a cold mixture of 3.6 milliliters of 50% potassium hydroxide and 17 milliliters of ether. After a few minutes the yellow ether layer of the mixture was decanted, dried over potassium hydroxide, and then added to a solution of 0.30 gram FES in 17 milliliters of ether. The resulting yellow mixture was left overnight in a loosely stoppered flask and then ether and diazomethane were evaporated using a steam bath. The remaining gummy residue was crystallized by adding 3 milliliters of water, heating to 60°C., and adding ethanol almost to solution.

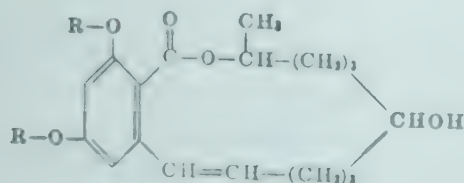
On cooling, crystals formed yielding 0.137 gram of a product having a melting point of 111° to 116°C. which was recrystallized in the same way to yield 0.082 gram of monomethyl FES having a melting point of 120° to 122°C. and analyzing:

|                                | Calc.<br>(C <sub>19</sub> H <sub>24</sub> O <sub>3</sub> ) | Found |
|--------------------------------|--|-------|
| Percent C.....                 | 68.7   | 68.3  |
| Percent H.....                 | 7.28   | 7.38  |
| Percent OCH <sub>3</sub> ..... | 9.34   | 9.17  |

The p-methyl FES is substituted for the dihydro FES in following essentially the same procedure used in Example 1 to produce a compound having the formula:



The compounds described in U.S. Patent 3,239,348; March 8, 1966, have the formula:



wherein R is selected from the group consisting of hydrogen and lower alkyl.

**Example 3 — FES Alcohol:** Sodium borohydride (1 gram) was slowly added to 50 milliliters methanol and 0.3 gram FES while cooling the resultant reaction mixture. The mixture was heated for 2 hours on a steam bath and the methanol is evaporated. The residue is neutralized

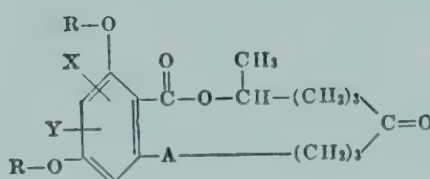


## Estrogens as Growth Stimulators

with HCl and extracted with two 40 milliliter portions of ether. The ether was evaporated and the residue dissolved in 10 milliliters isopropyl alcohol. After addition of 5 milliliters water and partial evaporation, the product, 0.21 gram, melted at 91° to 106°C. The FES alcohol hydrate product was recrystallized, was found to have a melting point of 90° to 102°C. and analyzed:

|                | Calculated | Found |
|----------------|------------|-------|
| Percent C..... | 63.88      | 62.98 |
| Percent H..... | 7.74       | 7.71  |

The compounds described in U.S. Patent 3,239,349; March 8, 1966, have the formula:



wherein A is a radical selected from the group consisting of  $-\text{CH}=\text{CH}-$  and  $-\text{CH}_2-\text{CH}_2-$ ; X is iodine; Y is selected from the group consisting of hydrogen and iodine and R is selected from the group consisting of hydrogen and lower alkyl.

Example 4 — Diiodo FES: Stock solution A was made by dissolving 433 milligrams of FES in 80 milliliters of acetic acid and diluting the resulting mixture to 100 milliliters with water. Stock solution B was made by dissolving 5 grams of periodic acid ( $\text{HIO}_4$ ) in 200 milliliters of water and adding 800 milliliters of acetic acid to the resulting solution.

To 20 milliliters of stock solution A was added 50 milliliters of stock solution B. After one-half hour, 3 milliliters of 20% KI solution was added and after an additional 16 hours, an additional 7 milliliters of KI solution was added. The excess iodine was titrated with thio-sulfate solution and the solid was collected by filtration, washed with water and dried in a vacuum desiccator. The product, in an amount of 91 milligrams and having a melting point of 88° to 96°C., was recovered and recrystallized first from cyclohexane and then from n-hexane to yield the diiodo FES product having a melting point of 125.5° to 127°C. and analyzing:

|                | Calc.<br>( $\text{C}_{19}\text{H}_{19}\text{O}_3\text{I}_2$ ) | Found |
|----------------|---|-------|
| Percent C..... | 37.91   | 37.66 |
| Percent H..... | 3.54  | 3.56  |
| Percent I..... | 44.52   | 43.67 |

Example 5 — Monomethyl and Dimethyl Diiodo FES: Dimethyl sulfate (5 milliliters) was added to a solution of 2.24 grams FES in 80 milliliters of a 10% NaOH solution and 20 milliliters of water. The mixture was stirred for one-half hour at 18° to 20°C. (cooling bath) and an additional 5 milliliters of dimethyl sulfate was added. After an additional 70 minutes of

## Estrogens as Growth Stimulators

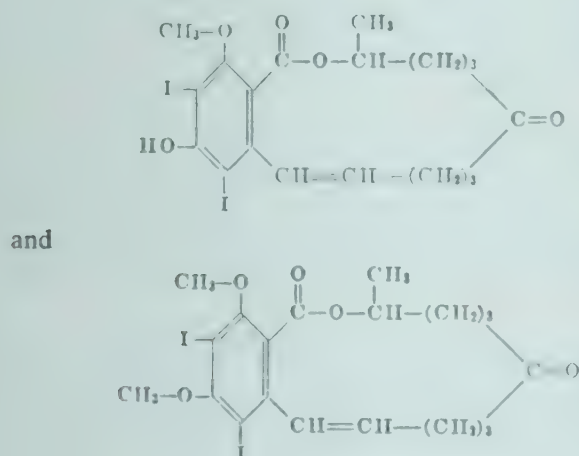
stirring at 20° to 26°C., the solid precipitate, solid A, was collected by filtration, washed with water and dried. The filtrate from solid A was acidified with 25 milliliters 12 N H<sub>2</sub>SO<sub>4</sub> to yield a second precipitate, solid B, which was collected, washed with water, and dried.

Solid A (0.79 gram having a melting point of 114° to 118°C.) was recrystallized from a mixture of 10 milliliters water and 15 milliliters ethanol to yield 0.66 gram of dimethyl FES having a melting point of 108° to 110°C.

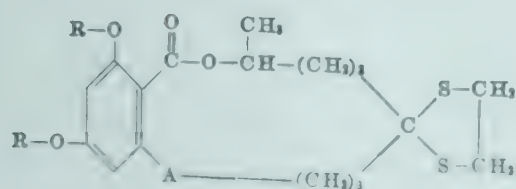
Solid B (1.39 grams having a melting point of 152° to 162°C.) was recrystallized twice from a mixture of water and alcohol to yield 0.80 gram of monomethyl FES product having a melting point of 169° to 174°C. Analysis of solid B showed:

|                  | Calc.<br>(C <sub>19</sub> H <sub>18</sub> O <sub>3</sub> ) | Found |
|------------------|--|-------|
| Percent C.....   | 68.65  | 67.97 |
| Percent H.....   | 7.28   | 7.16  |
| Percent OMe..... | 9.34   | 9.28  |

Each of the monomethyl FES and dimethyl FES is subjected to essentially the same iodine treatment described in Example 4 to produce the respective compounds:



The compounds described in U.S. Patent 3,373,024; March 12, 1968, have the formula:



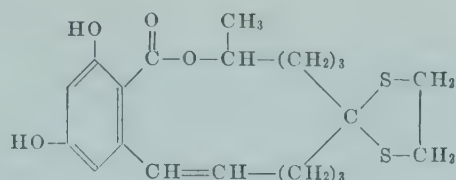
where A is selected from the group consisting of  $-\text{CH}=\text{CH}-$  and  $-\text{CH}_2\text{CH}_2-$  and R is selected from the group consisting of hydrogen, lower alkyl and lower saturated acyclic acyl.

**Example 6 — Ethylene Thioketal:** FES in an amount of 2 grams is refluxed in 100 cc benzene containing 2 milliliters of 1,2-ethane dithiol and 0.2 gram paratoluene sulfonic acid as an acidic catalyst for 2 hours and thereafter cooled to room temperature and washed with a 5% solution of sodium bicarbonate. Excess benzene and 1,2-ethane dithiol are distilled



## Estrogens as Growth Stimulators

off and the residue dissolved in boiling isopropyl alcohol. Upon cooling and addition of a small amount of water the product



precipitates out.

In a like manner monomethyl FES (Example 5) or dimethyl FES (Example 5) can be used instead of FES and reacted with 1,2-ethane dithiol to obtain other active derivatives. A mixture of alfalfa hay and ground corn cobs containing from 1 to 20 ounces of these estrogenic compounds per hundred pounds of ration were fed to cattle to increase their rate of growth.

Example 7: This is an example of an animal feed composition useful for increasing the rate of growth and feed efficiency of young animals to market weight. For young beef cattle, i.e., calves to yearlings running to two year olds, each animal is given 5 to 20 milligrams per day of the compound produced in Example 6 intimately admixed in about 18 to 22 pounds per head per day of a complete pelleted ration for about 180 days. The complete pelleted ration includes in addition to the compound of Example 6 the following:

|                          | <u>Percent</u>    |
|--------------------------|-------------------|
| Barley                   | 40 - 43           |
| Molasses dried beet pulp | 34.5 - 37.5       |
| Alfalfa pellets          | 8.0               |
| Tallow                   | 2.5               |
| Calcium carbonate        | 0.30              |
| Urea                     | 0.30              |
| Phosphorus source        | 0.40              |
| Salt                     | 0.50              |
| Molasses                 | 10.00             |
| Trace mineral            | 0.5               |
| Vitamin A                | 2 to 4 MMI.U./ton |

Note: Milo or corn, for example, can be substituted for the barley.

The compound of Example 6 is admixed with the above ingredients in a stationary blender or a feed mix truck in the following amounts in grams per ton to provide an appropriate complete pelleted feed with dosage levels ranging from 5 to 90 milligrams per head per day.

| <u>Grams/ton:</u> | <u>Mg./head/day</u> |
|-------------------|---------------------|
| .5 -----          | 5                   |
| 1.0 -----         | 10                  |
| 2.0 -----         | 20                  |
| 4.0 -----         | 40                  |
| 8.0 -----         | 80                  |

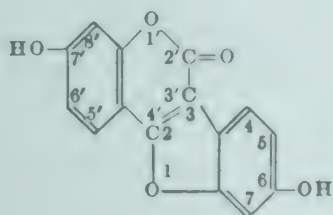
## Estrogens as Growth Stimulators

These gram amounts are premixed with, for example, 10 pounds of soybean hulls prior to admixture with the other ingredients.

### COUMARONE COMPOUNDS

#### Coumestrol Ethers

E.M. Bickoff and A.N. Booth (U.S. Patent 2,987,398; June 6, 1961; assigned to the U.S. Secretary of Agriculture) have a process for the preparation and use of coumestrol ethers. Coumestrol structurally is 7',6-dihydroxycoumarino(3',4'-3,2)-coumarone having the formula:



Example — Preparation: A mixture of 50 mg. coumestrol, 250 ml. acetone, and 2 g. potassium carbonate was refluxed on the steam bath under an atmosphere of nitrogen and while applying mechanical agitation. Dimethyl sulfate was added dropwise to the refluxing system until a total of 4 ml. had been added. After 45 minutes the reaction was completed and the reaction mixture was made alkaline with 6 N sodium hydroxide to hydrolyze residual dimethyl sulfate. The reaction mixture was then acidified with 6 N hydrochloric acid and the solids were filtered off and dissolved in 150 ml. acetone. The acetone solution was reduced to dryness under vacuum and the residue was recrystallized from methanol. The product, coumestrol dimethyl ether, was obtained in a yield of 48 mg. and had a melting point of 200° to 201°C. The product may also be termed 7',6-dimethoxycoumarino(3',4'-3,2)-coumarone.

It is obvious that by selection of the etherifying agent any desired ether of coumestrol can be synthesized. For example, the use of methyl bromide or dimethyl sulfate will yield the methyl ether, the use of ethyl bromide or diethyl sulfate will yield the ethyl ethers.

The coumestrol ethers may be employed in animal husbandry in the same manner that diethylstilbestrol and other known estrogenic agents are used. Thus the ethers may be administered by incorporating them in conventional feeds; by addition to water or other fluid; by addition to grit fed to birds; by administration in capsules, pellets or by injection; by implantation of pellets, and so forth. The amount of the ether to be administered will of course vary depending on the type of animal, the body weight thereof, the physiological response desired, and the mode of administration. For example, where the ether is administered in admixture with a feed, dosage may be that physiologically equivalent to about from 0.01 to 8 milligrams of diethylstilbestrol per 100 pounds of body weight per day. Generally it is preferred to administer the ether by incorporating it in a conventional feed.

In general the feed may contain on the order of 0.001 to 1 pound of coumestrol ether per ton of feed. The coumestrol ethers may be applied, for example, to chickens, turkeys, geese,



ducks, swine, sheep, cattle, horses, and so forth. Thereby, important practical effects are gained including increased rate of gain and increased efficiency of feed utilization.

### Other Coumarone Derivatives

In this process E.M. Bickoff, A.L. Livingston and A.N. Booth (U.S. Patent 3,077,404; February 12, 1963; assigned to the U.S. Secretary of Agriculture) describe the preparation of salts of 2-(2,4-dihydroxyphenyl)-6-hydroxy-coumarone-3-carboxylic acid. These have estrogenic activity and can be used in animal feeds as described above for coumestrol ethers.

**Example 1 — Preparation:** Twenty-seven parts of coumestrol was dissolved in about 1,000 parts of 1% KOH in methanol. The solution was warmed for 3 minutes on a steam bath. The resulting solution of the potassium salt of 2-(2,4-dihydroxyphenyl)-6-hydroxy-coumarone-3-carboxylic acid may be used as a source of said compound or it may be evaporated under vacuum to obtain the salt in solid form. The estrogenic activity of the coumarone derivatives of the process is illustrated by the following example.

**Example 2 — Estrogenic Activity:** Estrogenic assays were conducted by feeding one lot of immature female mice with a basal ration containing the potassium salt of 2-(2,4-dihydroxyphenyl)-6-hydroxy-coumarone-3-carboxylic acid. Another lot of the mice were fed the basal ration containing coumestrol. Each lot of mice contained 5 animals. In these tests each mouse was supplied with 10 grams of basal ration containing 0.3 or 0.75 mg. of the test compound and when this feed had been completely consumed (5 to 6 days) the feeding period was complete. A control lot of 5 animals were fed 10 grams of the basal ration.

After the feeding period was completed, the animals were sacrificed and their uteri were excised and weighed. An increase in uterine weight denotes estrogenic activity in the material under test, the greater the increase in uterine weight over the control, the more potent the material tested. The results of the assays are set forth below.

| Material Tested   | Amount Fed, mg. per mouse | Average Uterine Weight, mg. |
|---|---------------------------|-----------------------------|
| Potassium salt of 2-(2,4-dihydroxyphenyl)-6-hydroxycoumarone-3-carboxylic acid..... | 0.75                      | 86.5                        |
| Do.....   | 0.3                       | 27.0                        |
| Coumestrol.....   | 0.75                      | 90                          |
| Do.....   | 0.3                       | 25                          |
| Control (basal ration).....   |                           | 9                           |

### DIETHYLSTILBESTROL (DES) PREMIX

#### Compact Diethylstilbestrol Premix

F.X. Gassner and W.N. McLellan (U.S. Patent 2,960,407; November 15, 1960; assigned to Olin Mathieson Chemical Corporation) found that diethylstilbestrol could be accurately incorporated in emulsified fat as well as in molasses and the like, provided the diethylstilbestrol was dissolved in a liquid polyethylene glycol-acidulated fatty oil solution and that the resulting emulsions could be readily applied by spraying directly to the oilmeal or other

## Estrogens as Growth Stimulators

like feed. The diethylstilbestrol-polyethylene concentrate premix employed in this process, instead of involving the handling and storage of 0.5 to 1.0 ton of premix per 1,000 gram of diethylstilbestrol as found necessary heretofore, proved to involve only the handling and storage of about 6 lbs. of premix per 1,000 gram of diethylstilbestrol. This is accomplished without increasing mixing problems, and the savings in freight charges and storage to the feed mill are obvious. It also entails the ultimate obtainable in safety to the operators or feed processors, e.g., there are no dust problems and the wearing of masks and rubber gloves required heretofore by law becomes unnecessary.

### Example: Specific method — Step 1:

|                                |            |
|--------------------------------|------------|
| Crystalline diethylstilbestrol | 2,500 gram |
| Polyethylene glycol (200)      | 2,500 gram |

The diethylstilbestrol is dissolved in the liquid polyethylene glycol by heating the mixture to 85°C. with stirring. This mixture on cooling to room temperature forms a stable semi-solid white paste. This is the form in which it is shipped to the feed manufacturers.

Step 2: The paste obtained in Step 1 is liquefied by heating to 85°C. and is then poured with agitation into 192.3 liters of acidulated coconut oil containing about 2% free fatty acid and heated to about 50°C.

Step 3: Two liters of the diethylstilbestrol-polyethylene glycol-oil composition prepared as in Step 2 is then thoroughly mixed by agitation with 1,300 lbs. of hot fats heated at about 50°C.

Step 4: The composition prepared as in Step 3 is then added to a mixture made up of about 420 gallons of water and 6 gallons of 20% sodium hydroxide and the resulting mixture emulsified with vigorous agitation. The composition produced as above contains about 52 grams of the original diethylstilbestrol-polyethylene composition or 26 grams of diethylstilbestrol. The water-fat ratio is about 2.5:1 with about 1.5% caustic soda based on the fat.

The composition prepared as above containing about 1,300 lbs. of fat and 26 grams of diethylstilbestrol is then power sprayed with mixing on 20 tons of oilmeal or other feed. This provides a daily ration of 0.5 lb. of fat and 10 mg. of diethylstilbestrol for 2,600 steers and can be mixed with the final ration in accordance with standard practices in the art. The process is relatively simple and safe to the operators and analyses have shown the product to be homogeneous and proper dosage easily controlled.

The polyethylene glycol-diethylstilbestrol-acidulated fatty oil composition prepared as in Step 2 above, can be advantageously used in other ways besides in emulsion form as described in Steps 3 and 4. For example, the acidulated fatty oil composition can be merely mixed with various diluents such as vegetable oils including rice bran oil, soybean oil, corn oil, etc., as well as animal oils or mixtures of the same including crude oil foats, fatty by-products, etc. or be emulsified in beet or cane molasses, and be sprayed on cattle rations of various types used in the finishing of meat producing cattle.



## Improving Stability of Diethylstilbestrol-Containing Premix

A diethylstilbestrol-containing premix having greatly improved stability under storage conditions, which remains stable even in the presence of high levels of mineral supplements, has been developed by W.L. Bender and N.H. Ludwig (U.S. Patent 3,356,504; December 5, 1967; assigned to Eli Lilly and Company).

They found that diethylstilbestrol premixes with surprisingly improved stability result when a glycolic solvent vehicle is employed to distribute the diethylstilbestrol on the solid carrier. Because of its ready availability, lack of toxicity and desirable physical properties, an especially preferred solvent is propylene glycol. The table below shows the results of diethylstilbestrol potency determinations after eight weeks' storage of cattle feed compositions containing a high level of minerals: potency is expressed as a percentage of the initial diethylstilbestrol content.

Effect of Solvent Vehicle on Diethylstilbestrol Potency in Feed Premixes

| Solvent               | Carrier          | Storage Temperature |        |           |
|-----------------------|------------------|---------------------|--------|-----------|
|                       |                  | 25° C.              | 37° C. | 45-55° C. |
| Soybean oil, refined  | Soybean feed     | 26                  | 20     | 10        |
| Vegefat <sup>a</sup>  | Alfalfa granules | 66                  | 65     | 48        |
| P-200 <sup>b</sup>    | Soybean feed     | 75                  |        | 70        |
| P-2000 <sup>c</sup>   | Alfalfa granules | 82                  | 85     | 64        |
| PEG 20M <sup>d</sup>  | Soybean feed     | 94                  |        | 37        |
| PEG 6000 <sup>e</sup> | Soybean feed     | 93                  |        | 51        |

<sup>a</sup> A vegetable oil methyl ester.

<sup>b</sup> Polypropylene glycol having an average molecular weight of about 200.

<sup>c</sup> Polypropylene glycol having an average molecular weight of about 2,000.

<sup>d</sup> Polyethylene glycol having an average molecular weight of about 20,000.

<sup>e</sup> Polyethylene glycol having an average molecular weight of about 6,000.

Example 1: A steam-jacketed make-up tank is charged with about 440 liters of propylene glycol, the agitator is started, and live steam is admitted into the jacket. When the temperature of the propylene glycol reaches about 80°C., 11.25 kg. of diethylstilbestrol are added and stirring and heating are continued until solution results. Heating is discontinued and the diethylstilbestrol solution is added slowly to a mixer that has been charged with 4,536 kg. of solvent-extracted soybean meal. After further mixing for about an hour, the premix is ready for bagging or for incorporation into a high-mineral feed supplement.

Example 2: A heated make-up tank is charged with 64.356 kg. of PEG 6000 (a polyethylene glycol having an average molecular weight of about 6,000) and heat is applied until the internal temperature reaches about 80°C. The agitator is started and 10.726 kg. of diethylstilbestrol are added. When solution is complete, the molten solution is discharged into shallow trays which are kept at room temperature until solidification occurs. The resulting solid cake is ground in a hammermill to a degree permitting the particles to pass through a 20-mesh sieve. The ground material comprising the diethylstilbestrol coated with PEG 6000 is mixed for about an hour in a vertical mixer with sufficient soybean feed to produce a total batch size of 10,500 pounds. When thoroughly blended, the premix is suitable for bagging or for incorporation into a high-mineral feed supplement or a complete feed ration.

## Safe Handling of Estrogenic Feed Supplements

The widespread use of a very potent estrogen has given rise to a serious problem of handling feed supplements. It is well known that diethylstilbestrol and other estrogens are absorbed through the skin and the respiratory tract, and when so absorbed can produce secondary female sex characteristics in males. In the chemical and pharmaceutical industries, rigorous precautions are observed to avoid contact with such materials. In the animal feed industry, however, there is much more danger of contact because of the nature of the personnel and the lack of scientific control.

L. Rosner and R.O. Foster (U.S. Patent 2,930,695; March 29, 1960; assigned to Rosner-Hixson Laboratories, Inc.) have a process to provide an animal feed ingredient which is physiologically effective when ingested and which is safe for humans to handle, by having the estrogen in a readily assimilable gelled vehicle wherein the active constituent is shielded from contact with humans.

The assimilable gel may be any nontoxic water-soluble colloid which is nontoxic for animal consumption. It may be natural material such as gelatin, agar, alginates or pectin, or a synthetic low molecular weight polymer such as polyvinyl alcohol or polyvinylpyrrolidone. It may also be a denatured material such as a cellulose ether, including methylcellulose and carboxymethylcellulose. It is usually desirable to add nontoxic binders or plasticizers to the vehicle to improve the characteristics of the gel and to aid in the dispersion of the active ingredients. Suitable for these purposes are polyhydroxy organic compounds including sugars and derivatives thereof, such as corn syrup, glycerol, mannitol, sorbitol, glucose, sucrose, dextran and dextrans. These materials are preferably added in the form of syrups, usually in water.

Example 1 — Preparation: Four-hundred milligrams of diethylstilbestrol was mixed by mortaring with 100 grams of starch. Two-hundred and twenty milliliters of water and 25 grams of corn syrup were added to the mixture and heated in a boiling water bath until the starch had gelatinized. The hot material was spread on nylon cloth and allowed to air dry for 24 hours. The nylon sheet, with the material, was folded and placed in a 60°C. oven for 24 hours. The starch material was removed from the nylon and milled to a suitable particle size, placed in 2 liters of ether and stirred for one hour. The material was removed by filtering and air dried. A yield of 87 grams of powder assaying 1.7 milligrams of diethylstilbestrol per gram was obtained.

Example 2 — Handling: The materials to be tested were applied by adhesive bandages to the backs of weanling female rats for a period of three days. The animals were then sacrificed, the uteri removed, and the average weight of the uteri computed and compared to the uterine weight of an untreated control group. Typical results of skin absorption tests are given in the following table:

| Supplement | Dilution | Diethylstilbestrol Assay, micrograms per 100 milligrams | Average Uterine Weight, milligrams (for 5 rats) |
|------------|----------|---|---|
| Commercial | 1:20     | 11  | 62.8  |
| Example 1  | 1:20     | 8.8   | 11.9  |
| Example 1  | none     | 176   | 14.2  |
| Control    |          | 0   | 18.0  |

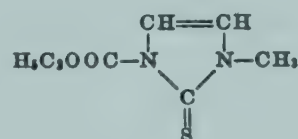


These data demonstrate that the compositions of this process completely protect against absorption of physiologically active materials through the skin, whereas the largest selling commercial preparation, even when diluted 20 to 1 with inert meal, allows very substantial absorption of diethylstilbestrol through the skin.

## SYNERGISTIC EFFECT OF DES

### Estrogen Plus 1-Methyl-2-Mercapto-3-Carbethoxy Imidazole

B.J. Brent (U.S. Patent 2,977,230; March 28, 1961; assigned to The Denver Chemical Manufacturing Company) found it possible to increase the live weight and to improve the meat quality of meat producing animals as well as to effect better feed utilization, by feeding to such animals 2-mercapto imidazole compounds. An especially suitable compound of this type is the 1-methyl-2-mercapto-3-carbethoxy imidazole.



The optimum amount of active agent to be orally administered is regulated according to the weight and the food consumption of the animal. It was found that, when using 1-methyl-2-mercapto-3-carbethoxy imidazole, amounts between about 20 mg. and 80 mg. per day per 100 lbs. of live weight added to the feed supplement or to the feed ration consumed by the animal, resulted in satisfactory weight gains and improvement of meat quality.

Hereford heifers were used for fattening. 1-methyl-2-mercapto-3-carbethoxy imidazole was fed to such heifers in doses of 500 mg. per day for 35 days. It is clearly evident that administration of the active agent, at the 500 mg. level, causes a considerable gain in weight and decreases the amount of feed required for the gains. The main increase in gain took place after the first 20 day period. The daily gain on treatment with 1-methyl-2-mercapto-3-carbethoxy imidazole remains constant during the first and second period.

Example: To prove the surprising synergistic effect of a combined feeding of 1-methyl-2-mercapto-3-carbethoxy imidazole and DES, comparative tests were carried out. The following table shows the weight increase in percent observed when administering 1-methyl-2-mercapto-3-carbethoxy imidazole alone, DES alone, and a mixture of 1-methyl-2-mercapto-3-carbethoxy imidazole and stilbestrol. 0.2% of the active agents were admixed to the feed. The following table shows the weight gains over the controls.

|              | 1-Methyl-2-mercapto-3-carbethoxy imidazole, 0.2% | 1-Methyl-2-mercapto imidazole, 0.2% | Stilbestrol, 0.2% | 1-Methyl-2-mercapto-3-carbethoxy imidazole, 0.2%+ Stilbestrol, 0.2% | 1-Methyl-2-mercapto imidazole, 0.2%+ Stilbestrol, 0.2% |
|--------------|--|-------------------------------------|-------------------|---|--|
|              | Percent  | Percent                             | Percent           | Percent   | Percent  |
| 1st week---- | -4.1   | -2.0                                | +2.0              | +2.0  | +3.1   |
| 2nd week---- | +7.0   | +7.8                                | +9.6              | +8.7  | +8.7   |
| 3rd week---- | +9.3   | +9.3                                | +13.2             | +19.4   | +16.3  |
| 4th week---- | +15.7  | +12.9                               | +13.6             | +20.4   | +15.7  |

The toxicity of 1-methyl-2-mercapto-3-carbethoxy imidazole is remarkably low. Dogs, which were fed 1 mg./kg., 3 mg./kg., and 9 mg./kg. of 1-methyl-2-mercapto-3-carbethoxy imidazole did not show any noticeable pathological changes in the investigated organs, such as spleen, heart, gastro-intestinal tract, liver, kidney, pancreas, testes, brain and meninges, gall bladder, adrenals, lungs, ovaries, bladder. The bone marrow was found to contain large amounts of fat and a very good hematopoietic activity. The testes showed good spermatogenesis. It is evident that even with the above mentioned high doses given to dogs over a three month period, no noticeable toxic effects are caused by 1-methyl-2-mercapto-3-carbethoxy imidazole.

### Estrogen Plus 2-Mercaptoimidazole

W. Burroughs and T.M. Means (U.S. Patent 3,041,173; June 26, 1962; assigned one-half to Iowa State College Research Foundation and one-half to Eli Lilly and Company) found growth promoting properties for beef cattle and sheep fed a composition containing estrogenic substance and a 2-mercaptoimidazole compound, the preferred composition being diethylstilbestrol combined with 1-methyl-2-mercaptoimidazole.

The major benefits of this process are achieved over feeding periods ranging from 30 to 90 days. The most pronounced enhancement of the estrogenic growth response occurs during the first 30 days of the combined feeding of the estrogen and the 2-mercaptoimidazole compound, and there will usually not be any great advantage to extending the combined feeding beyond 90 days. The method is therefore particularly adapted for use in the final months of feeding of beef cattle prior to marketing.

Example 1: 10 grams of diethylstilbestrol is dissolved in 227 grams of soybean oil. The oil solution of the estrogen is then thoroughly mixed with 8.5 pounds of dry, oil-free soybean meal. The resulting material is next thoroughly mixed and blended with 500 grams of 1-methyl-2-mercaptoimidazole which is in the form of a fine dry powder. When the 2-mercaptoimidazole compound has been distributed throughout the soybean meal material, the resulting premix product will contain 50 grams of the 2-mercaptoimidazole compound and 1 gram of diethylstilbestrol per pound of the product.

The premix product prepared as described above (which contains both the diethylstilbestrol and the 1-methyl-2-mercaptoimidazole in the proportions of 50 parts of the 2-mercaptoimidazole compound per part of the diethylstilbestrol) can then be used to prepare a feed ration or protein supplement for administration to beef cattle. For example, 10 pounds of the premix can be mixed with 1,990 pounds of a beef cattle protein supplement. Such supplements are composed principally of a high protein component from a vegetable source such as soybean meal, cottonseed meal, linseed meal, wheat bran, etc. After the premix has been thoroughly distributed throughout the protein supplement, a ton of a feed material utilizable in accordance with this process will be obtained. This material will contain 5 milligrams of diethylstilbestrol per pound and 250 milligrams of 1-methyl-2-mercaptoimidazole per pound, and is well adapted for use as finishing-type protein supplement in the feeding of beef cattle for market.

Example 2: In practicing the method of this process it is preferred to utilize steers, although the method is also applicable to heifers. By way of specific example, yearling steers weighing



an average of 700 pounds can first be fed for ninety days on a ration which includes approximately 10 milligrams of diethylstilbestrol per day per animal. At the end of that period the steers have average weights of say 950 pounds. At this point, the feeding of the finishing ration will be started. This ration will include the special protein supplement described in Example 1, which will be fed at the rate of 2 pounds per day per animal for a further period of sixty days. This will mean that the animals will be receiving approximately 10 milligrams of diethylstilbestrol and 500 milligrams of 1-methyl-2-mercaptoimidazole per 24 hours. The rate of weight gain and the feed efficiency will be substantially higher during this 60 day period than in the preceding 90 day period when the diethylstilbestrol was employed alone. The weights of the animals at the end of the final 60 day feeding period might average around 1,150 to 1,200 pounds.

### Estrogen Plus Steroidal Sapogenin

J.E. McKeen and W.J. Haas (U.S. Patent 3,144,337; August 11, 1964; assigned to Chas. Pfizer & Co., Inc.) found that when an estrogenic substance is concurrently administered to an animal together with a steroidal sapogenin, there is obtained an even greater growth response than could possibly be anticipated from the use of the sapogenin or estrogenic substance alone. The sapogenins employed are nontoxic in nature and they are preferably selected from the group consisting of smilagenin, sarsasapogenin and hecogenin. Diethylstilbestrol is the preferred estrogenic substance.

A level as low as 0.1 gram of one of the aforesaid sapogenins per ton of animal feed is sufficient to impart a substantial growth response to the animal. In general, the nontoxic steroidal sapogenin can be employed in feeds at concentration levels ranging from about 0.1 gram to about 24 gram per ton of feed in order to obtain the unusually high degree of growth promotion.

Example: Lambs were fed on a nutritious diet. The results obtained after 56 days of treatment are summarized below in the following table.

| Treatment  | No. of lambs | Av. daily gain (lb.) | Percent increase | Feed per lb. gain |
|--|--------------|----------------------|------------------|-------------------|
| Control .....  | 24           | 0.368                | -----            | 6.84              |
| Smilagenin, 8.0 g./ton.....                                  | 12           | 0.390                | 5.8              | 6.23              |
| Diethylstilbestrol, 1.25 g./ton..                            | 12           | 0.432                | 17.4             | 6.13              |
| Diethylstilbestrol, 1.25 g./ton and smilagenin, 8.0 g./ton.. | 12           | 0.534                | 45.1             | 5.25              |

In addition treated animals show an improved carcass grade.

### OTHER HORMONES

#### 9 $\alpha$ -Fluoro-16 $\alpha$ -Methylprednisolone

An increased rate of growth and feed utilization from the administration of 9 $\alpha$ -fluoro-16 $\alpha$ -methylprednisolone to feed was found by L.D. Harrop (U.S. Patent 3,036,917; May 29, 1962; assigned to The Upjohn Company). The concentration of steroid in the feed composition

## Estrogens as Growth Stimulators

is determined with regard to the weight of the animal and the average amount of feed consumed daily. The concentration is then that amount which will provide up to 1 mg. of 9 $\alpha$ -fluoro-16 $\alpha$ -methylprednisolone per 100 lb. of body weight per day. The daily consumption of from 0.001 to 1 mg. of steroid per 100 lb. of body weight is preferred.

A group of fifty weaned Chester-White pigs, approximately ten weeks of age, were weighed individually and then randomly allotted among five groups on the basis of weight and sex. Each group of ten pigs was placed in a separate concrete floored pen, provided with an automatic waterer and self-fed a diet as shown in Table 1.

TABLE 1:

| Group  | Diet   |
|--------|--|
| 1..... | Swine Growing Diet.  |
| 2..... | Swine Growing Diet with 9 $\alpha$ -fluoroprednisolone acetate, 0.8725 mg./lb. feed.             |
| 3..... | Swine Growing Diet with 9 $\alpha$ -fluoroprednisolone acetate, 1.715 mg./lb. feed.              |
| 4..... | Swine Growing Diet with 9 $\alpha$ -fluoro-16 $\alpha$ -methylprednisolone, 0.1725 mg./lb. feed. |
| 5..... | Swine Growing Diet with 9 $\alpha$ -fluoro-16 $\alpha$ -methylprednisolone 0.3430 mg./lb. feed.  |

The results of thirteen days' feeding is shown in Table 2.

TABLE 2:<sup>1</sup>

| Group (10 pigs/group)                             | 1     | 2     | 3     | 4     | 5     |
|---|-------|-------|-------|-------|-------|
| Starting Wt., lb.....                             | 61.2  | 61.9  | 61.4  | 61.9  | 61.5  |
| Final Wt., lb.....                                | 75.8  | 77.1  | 74.1  | 79.2  | 77.2  |
| Daily Gain/pig.....                               | 1.12  | 1.17  | 0.98  | 1.33  | 1.21  |
| Daily Feed Intake/lb. <sup>2</sup> .....          | 3.4   | 3.8   | 3.6   | 3.1   | 3.4   |
| Lb. Feed Consumed/100 lb. gain <sup>2</sup> ..... | 301.4 | 323.7 | 364.6 | 235.8 | 278.3 |

<sup>1</sup> Figures represent averages for each group.

<sup>2</sup> Feed wastage occurred in all groups and observations indicated that the degree of feed wastage was similar for each of the groups. Therefore, while the figures pertaining to feed consumption and feed utilization are a little high, the relative values are correct.

Example: A fattening feed for 800 pound yearling cattle is prepared from the following types and amounts of ingredients:

|                       |        |                                    |       |
|-----------------------|--------|------------------------------------|-------|
| Ground ear corn       | 89.75% | Salt                               | 0.5 % |
| Soybean oil meal, 44% | 9.0 %  | Trace mineral mixture <sup>1</sup> | 0.05% |
| Ground limestone      | 0.7 %  |                                    |       |

<sup>1</sup> Contains the following percent of minerals: Mn, 12; Co, 0.08; Fe, 5.0; Cu, 0.4; I, 0.24; Zn, 0.7.



## Estrogens as Growth Stimulators

To 99 parts of the preceding feed is added 1 part of a premix composition prepared by mixing 12.9 mg. of 9 $\alpha$ -fluoro-16 $\alpha$ -methylprednisolone with sufficient wheat flour to make 1 pound. The feeding composition so prepared supplies 0.129 mg. of steroid per pound, or about 1 part in 4 million. Cattle are to receive the foregoing feed ad libitum together with 5 lb. of hay per head per day and when so fed have an increased rate of weight gain and improved utilization of feed.

### Glucocorticoids

J. R. De Zeeuw and F. Sauer (U.S. Patent 2,951,759; September 6, 1960; assigned to Chas. Pfizer & Co., Inc.) found that the addition of a low level of glucocorticoids to the balanced diet of various simple stomached (non-ruminant) animals such that these animals receive the product over an extended period of time, that is, a major portion of their active growth period, results in an acceleration of the rate of growth.

In general it is preferred to use the glucocorticoids at a concentration not greater than about 1 mg. of the compound per kg. of the total feed given to the animal. A concentration in general of at least about 0.001 mg./kg. is satisfactory. A range of about 0.01 mg./kg. of feed to about 0.1 mg./kg. seems to be the most effective concentration. Useful glucocorticoid hormones include hydrocortisone, cortisone,  $\Delta^1$ -dehydrocortisone,  $\Delta^1$ -dehydrohydrocortisone, 9-fluorohydrocortisone, 9-fluorocortisone, 14-hydroxyhydrocortisone and esters.

Example: Groups of baby chicks containing ten animals each were fed on standard highly nutritious broiler rations. In the following table is summarized the results of the gain in weight of the animals over a period of four weeks on these diets and animals of the same type and on the same diet supplemented with 0.10 mg. of hydrocortisone per kg. of feed.

| Level of hydrocortisone<br>(Mg./kg. diet) | Average<br>Weight<br>Gained<br>(20%<br>protein<br>diet), g. | Average<br>Weight<br>Gained<br>(20%<br>protein<br>diet), g. | Average<br>Weight<br>Gained<br>(22%<br>protein<br>diet), g. | Average<br>Weight<br>Gained<br>(22%<br>protein<br>diet), g. |
|---|---|---|---|---|
| 0.....                                    | 394   | 380   | 384   | 397   |
| 0.10.....                                 | 404   | 400   | 402   | 409   |

It is apparent from this tabulation that the addition of extremely low levels of hydrocortisone is effective in stimulating the growth of chickens.

### Mass Hormonization

W.E. Martox (U.S. Patent 3,042,525; July 3, 1962; assigned to Mattox and Moore, Inc.) provides a mass hormonization procedure in which the hormonizing drug is administered in the drinking water supplied to the animals, and especially to poultry; and thereby provides an improved and more reliable and effective uniform hormonizing action.

The hormonizing compound is administered in a concentration sufficient to produce characteristic hormonizing results over a suitable treatment period, of the order of from three weeks to thirty days. An estrogenic compound is desirably administered to chickens in a daily dose per chicken equivalent to about 0.3 mg. to about 2.0 mg. of diethylstilbestrol, and prefer-

## Estrogens as Growth Stimulators

ably equivalent to about 0.6 mg. to about 1.0 mg. of diethylstilbestrol. The desired dosage is thus in an amount such that each chicken will receive the equivalent of from 10 mg. to 50 mg., and desirably about 20 mg., of diethylstilbestrol over a thirty day treatment period.

The optimum dosage of 20 mg. of diethylstilbestrol itself per chicken over a thirty day period may be obtained as follows: The relatively water-insoluble diethylstilbestrol is dissolved in a liquid polyethylene glycol, such as the product available on the market as polyethylene glycol 200 and consisting of a mixture of polyethylene glycols having an average molecular weight of 200. The solution is desirably in a concentration of 2 grams of diethylstilbestrol in from 25 to 100 grams of polyethylene glycol 200. This solution is preferably first mixed with an aqueous alkaline solution containing alkalizing compounds which have a solubilizing effect for the diethylstilbestrol, which desirably have germicidal and sanitizing properties, and which are nontoxic in the concentrations which will occur in the final drinking water mixture.

The alkaline mixture desirably also includes one or more germicidal and sanitizing ingredients such as one of the quaternary ammonium salts available on the market for addition to poultry drinking water as a sanitizing agent, and which have solubilizing properties as wetting agents. The sanitizer and the alkalizing compounds may be mixed in concentrated solution with the polyethylene glycol solution, and this mixture then diluted to a predetermined volume, say a gallon, to form the concentrate for admixture with the drinking water.

Example: A flock of chickens was divided into six groups of approximately 100 birds each. One group was used as a control group and the other five groups were treated with different hormonizing mixtures or compounds in their drinking water. The chickens used were a White Rock breed sometimes referred to as "Cornish Cross" breed which is a standard meat-type bird. The birds were seven weeks old at the start of the hormonizing treatment. The results of the tests and scoring with the six groups of chickens are set forth in the following table.

|                                | Group 1 | Group 2 | Group 3 | Group 4 | Group 5 | Group 6 |
|--------------------------------|---------|---------|---------|---------|---------|---------|
| Number of birds.....           | 80      | 95      | 101     | 97      | 97      | 72      |
| cockerels.....                 | 43      | 43      | 53      | 43      | 40      | 37      |
| pullets.....                   | 37      | 52      | 48      | 54      | 57      | 35      |
| Average Weight—10 wks.:        |         |         |         |         |         |         |
| cockerels (lbs.).....          | 4.30    | 4.66    | 4.66    | 4.43    | 4.34    | 4.54    |
| pullets (lbs.).....            | 3.54    | 3.45    | 3.60    | 3.41    | 3.47    | 3.47    |
| Total Live Wt. (lbs.).....     | 316.0   | 379.8   | 420.5   | 375.0   | 371.5   | 289.5   |
| Total Feed—10 wks. (lbs.)..... | 824.7   | 978.0   | 1029.0  | 979.0   | 972.0   | 754.0   |
| Feed Conversion Ratio.....     | 2.61    | 2.58    | 2.44    | 2.61    | 2.62    | 2.60    |
| Production Efficiency.....     | 151     | 155     | 158     | 144     | 146     | 155     |
| Birds Dressed.....             | 50      | 50      | 101     | 97      | 50      | 72      |
| cockerels.....                 | 25      | 25      | 53      | 43      | 25      | 37      |
| pullets.....                   | 25      | 25      | 48      | 54      | 25      | 35      |
| Total Live Wt. (lbs.).....     | 201.0   | 198.5   | 420.5   | 375.0   | 199.0   | 289.5   |
| Eviscerated Wt. (lbs.).....    | 133.2   | 140.2   | 303.0   | 253.0   | 142.0   | 231.0   |
| Percent Yield.....             | 66.3    | 70.7    | 72.2    | 67.5    | 72.4    | 79.8    |
| Grading Scores:                |         |         |         |         |         |         |
| Breast Finish.....             | 55      | 55      | 75      | 55      | 55      | 80      |
| Vent Fat.....                  | 55      | 70      | 55      | 75      | 75      | 60      |
| Pigmentation.....              | 50      | 70      | 65      | 70      | 75      | 65      |
| Uniformity.....                | 50      | 55      | 75      | 80      | 90      | 75      |
| General Appearance.....        | 50      | 90      | 70      | 85      | 90      | 80      |
| Shanks.....                    | 75      | 75      | 75      | 75      | 75      | 75      |
| Wattles.....                   | 50      | 95      | 60      | 70      | 90      | 70      |
| Combs.....                     | 50      | 95      | 60      | 75      | 95      | 75      |
| Feathering.....                | 65      | 65      | 65      | 65      | 65      | 65      |



The treatments of the groups listed in the table were as follows:

- Group 1 — Control. No hormone. Sanitizer only.
- Group 2 — 20 mg. diethylstilbestrol di-(sulfoacetate). No sanitizer.
- Group 3 — 20 mg. diethylstilbestrol diphosphate. No sanitizer.
- Group 4 — 10 mg. diethylstilbestrol di-(sulfoacetate). With sanitizer.
- Group 5 — 50 mg. diethylstilbestrol di-(sulfoacetate). With sanitizer.
- Group 6 — 20 mg. diethylstilbestrol. With sanitizer.

The results obtained as set forth in the table establish that the chickens in each of groups 2 through 6 underwent characteristic hormonizing effects, and that the desired effects occurred with a high degree of uniformity on all the birds of each group. This is shown especially by the fact that graded characteristics were improved in high degree with the same or better "feed conversion ratios" and "production efficiency" indices; and by the valuable improvements in percent yield.

The tests lasted only 21 days instead of the 30 for which dosages were originally calculated, and the birds of groups 2 through 5 actually received only about two-thirds of the calculated 30-day dosage. The 72 birds of group 6 consumed the intended dosage for 100 birds, and this substantially offset the reduction in dosage resulting from the shortened treatment period, and the birds of group 6 thus received substantially the intended 20 mg. dosage over the three-week period.

The characteristic hormonization effects were obtained in high degree with total dosages actually ranging from only about 7 mg. of diethylstilbestrol equivalent in group 4, to 42 mg. in group 5. Taking into account the relative number of pullets to cockerels in the several groups, the results indicate that optimum results can be obtained with dosages of the order of 20 mg., and that larger dosages, while effective, are not necessary and merely increase the cost of medicament. The results thus indicate that this method of hormonizing by way of the drinking water is effective with dosages not far different from those which are effective by injection.

## ANTIBIOTICS AS ANABOLIC STIMULATORS

When antibiotics are incorporated into feeds, a marked stimulation in the growth of poultry and livestock is observed. There is better utilization of feed, increased bloom and appearance (carcass grade) control of disease (scours and enteritis) and a reduction in the number of runts. These processes are concerned with improving the stability of feeds containing antibiotics. In addition, growth factors found in new antibiotics and fermentation cultures are reported.

### STABILIZING BACITRACIN FEEDSTUFFS

Bacitracin may be prepared by the cultivation of microorganisms and particularly Bacillus subtilis. One difficulty with bacitracin is, however, that it is unstable under ordinary conditions of storage. Also, in the production of bacitracin by fermentation methods a considerable amount of the bacitracin may be lost during the normal process of recovery, which process usually involves the steps of evaporating and drum drying the fermentation mash.

#### Use of Manganese

R. A. Zorn (U.S. Patent 2,985,533; May 23, 1961; assigned to Grain Processing Corporation) found that an improved bacitracin-containing product may be prepared by adding thereto a small amount of a water-soluble compound of manganese. The manganese serves to stabilize the bacitracin against deterioration in storage.

Example: An aqueous fermentation medium was prepared containing 7.5 percent soybean flour, 2.0 percent cornstarch, 1.0 percent calcium carbonate, and 0.33 percent magnesium sulfate. This medium was sterilized by conventional techniques and was then inoculated with one-half volume percent of a 24-hour liquid culture of Bacillus subtilis. The inoculated medium was then incubated at about 30°C. under constant aeration conditions for a period of 30 hours. At the end of the fermentation period the pH was about 8.1.

After the fermentation had been completed, 1.0 percent by weight of manganese sulfate was added to the liquid fermentation mash and thereafter the fermentation product is suitable for sale to feed manufacturers as such or it may be standardized to a predetermined bacitracin potency utilizing corn meal, soybean meal, or other nutrient diluent. Thus, one suitable feed supplement is one in which about equal parts of drum-dried material and corn meal are blended to produce a product containing 10 grams of bacitracin per pound



of feed supplement.

### Manganese Plus Reducing Agent

R.A. Zorn, R.C. Malzahn and A.M. Hanson (U.S. Patent 2,985,534; May 23, 1961; assigned to Grain Processing Corporation) describe the preparation of an improved bacitracin containing product, by adding thereto a small amount of a water soluble compound of manganese and a small amount of a water soluble reducing agent. The aqueous fermentation is carried out as described above using a culture of Bacillus licheniformis.

After the fermentation has been completed, 1.0% by weight of manganese sulfate plus 1.0% by weight of sodium thiosulfate are added to and thoroughly mixed with the liquid fermentation mash. Thereafter the fermentation mash is concentrated in a conventional evaporator and then drum dried. The concentration of the bacitracin is typically 20 grams per pound of the dried product, suitable for use in feed supplement as above.

Example: In order to demonstrate the stability of the products, the following procedure was used to give a rapid evaluation: A one gram sample of a dried bacitracin-containing fermentation residue is placed in a screw-cap, plastic tape sealed 200 x 25 mm. tube and subjected to the heat of flowing steam (99°C.) for varying periods of time.

The steam chest stability of three bacitracin products at 5, 6, 7 and 8 percent moisture:

| Bacitracin product   | Moisture content, percent | Recovery after steaming |                  |                  |
|--|---------------------------|-------------------------|------------------|------------------|
|  |                           | 1 hour, percent         | 2 hours, percent | 3 hours, percent |
| Untreated control.....   | 5                         | 22.2                    | 12.2             | ca 4.3           |
|  | 6                         | 19.4                    | 9.4              | ca 5.1           |
|  | 7                         | 18.7                    | 11.0             | ca 7.4           |
|  | 8                         | 16.4                    | ca 7.3           | ca 4.4           |
| 1% MnSO <sub>4</sub> .....   | 5                         | 90.4                    | 76.5             | 73.9             |
|  | 6                         | 79.7                    | 59.0             | 62.5             |
|  | 7                         | 83.2                    | 57.5             | 33.8             |
|  | 8                         | 76.2                    | 52.4             | 35.4             |
| 1% MnSO <sub>4</sub> +1% Na <sub>2</sub> S <sub>2</sub> O <sub>4</sub> ..... | 5                         | 83.7                    | 79.7             | 71.2             |
|  | 6                         | 87.5                    | 83.8             | 71.2             |
|  | 7                         | 90.2                    | 79.9             | 70.3             |
|  | 8                         | 79.8                    | 73.6             | 65.1             |

### Use of Cobalt

In this process R.A. Zorn (U.S. Patent 3,021,217; February 13, 1962; assigned to Grain Processing Corporation) found that a small amount of a water-soluble compound of cobalt serves to stabilize the bacitracin against deterioration on storage. Again the aqueous fermentation is carried out as previously described with Bacillus subtilis.

After the fermentation had been completed, 1.0 percent by weight of cobalt sulfate was added to the liquid fermentation mash and thereafter the fermentation mash was concentrated, dried and utilized as described above. A characteristic of the drum-dried material and of the feed supplement is that the bacitracin contained therein is very stable under storage conditions.

Ligin-Bacitracin Complex

Stable bacitracin products are obtained by the process of J. Ziffer and T.J. Cairney (U.S. Patent 3,035,919; May 22, 1962; assigned to Pabst Brewing Company) by adding an alkali-soluble lignin protein precipitant to a solution of bacitracin.

The ligno-bacitracin compositions thus produced are evidently complexes, as follows from their exceptional stability to heat as shown below:

| Bacitracin preparation  | Initial activity (units per gram) | Percent activity remaining after indicated hours at 110°-112° C. |         |         |          |
|---|-----------------------------------|--|---------|---------|----------|
|   |                                   | 2 hours  | 4 hours | 6 hours | 24 hours |
| Pharmaceutical grade bacitracin (prepared from soluble Orzan A ligno-bacitracin)..... | 60,000                            | 52   | 49      | 45      | 20       |
| Dried bacitracin fermentation product.....  | 7,400                             | 32   | 12      | 12      | 5        |
| Soluble Orzan A lignobacitracin.....  | 11,100                            | 68   | 63      | 69      | 39       |
| Insoluble Orzan A ligno-bacitracin.....   | 10,300                            | 80   | 70      | 73      | 44       |
| Soluble Orzan S ligno-bacitracin.....   | 15,900                            | 84   | 72      | 66      | 52       |
| Soluble Toranil B ligno-bacitracin.....   | 14,900                            | 84   | 75      | 70      | 51       |

Example 1: Eighty grams of sodium lignosulfonate (Polyfon R) dissolved in 300 ml. of water, were added to 2 liters of bacitracin filtrate (pH 6.7) assaying 377 units per milliliter. The solution was then adjusted to pH 4.3 with 18 N sulfuric acid and the precipitated ligno-bacitracin complex recovered by centrifugation. The recovered solid was washed with pH 4.3 water (630 ml.). One-half of the washed solid was then slurried with enough water to form a thin paste and the slurry dried on a vacuum double drum rotary drier (29 inches vacuum, 15 lbs. per square inch steam pressure). 11.6 grams of water insoluble solid ligno-bacitracin composition, assaying 14,400 bacitracin units per gram, were obtained.

The remaining one-half of the washed solid was slurried in water to form a thin paste and the slurry adjusted to pH 7 with ammonium hydroxide, solubilizing the ligno-bacitracin complex. The solution was then dried using the above conditions. Sixteen grams of a water soluble solid, assaying 14,000 bacitracin units per gram, were obtained.

Example 2: Multi-stage precipitation produces initial fractions of higher potency. In the first precipitation stage, ammonium lignosulfonate (Orzan A) was added, in the amounts indicated in the table on the next page, to 2,000 ml. portions of bacitracin filtrate (pH 6.7) assaying 428 units per milliliter. The solutions were then adjusted to pH 3.5 with 20% NaHSO<sub>4</sub> and the precipitated ligno-bacitracin complexes recovered by settling and decantation at 40°C. The recovered supernatants were then used at pH 3.5 for the second precipitation stage. Orzan A was added to the supernatants as indicated in the table to follow. After readjustment of the mixtures to pH 3.5, the precipitated ligno-bacitracin complexes were recovered by settling and decantation at 40°C. The recovered solids from the two precipitation stages were solubilized by adjustment to pH 6.5 with 4N NH<sub>4</sub>OH.



## Antibiotics as Anabolic Stimulators

| Orzan A quantity,<br>percent by weight<br>of solution |                 | Bacitracin<br>activity<br>recovered<br>as ligno-<br>bacitracin<br>complex,<br>percent | Activity of<br>complex,<br>units per<br>gram on<br>dry basis |
|---|-----------------|---|--|
| First<br>stage  | Second<br>stage |   |  |
| 0.5   | -----           | 22.1  | 23,000   |
| -----   | 3.5             | 39.9  | 14,300   |
| 1.0   | -----           | 36.7  | 20,000   |
| -----   | 3.0             | 30.1  | 11,100   |
| 2.0   | -----           | 59.4  | 21,100   |
| -----   | 2.0             | 13.2  | 5,800  |
| 3.0   | -----           | 68.0  | 15,600   |
| -----   | 1.0             | 6.5   | 3,300  |
| 4.0   | -----           | 70.0  | 16,900   |
| -----   | 0.0             | -----   | -----  |

### Bacitracin Adsorbate (Cation Exchange Resin)

I. J. Friedman and E. G. Martin (U.S. Patent 3,074,795; January 22, 1963; assigned to Chas. Pfizer and Company Inc.) found that bacitracin is isolated from its solutions, including fermentation broths, by contact with certain sulfonated copolymers of styrene and divinylbenzene. The resins employed are remarkably selective for bacitracin and exhibit unprecedented capacity for the antibiotic.

These adsorbates are eminently suitable for use in feed supplements for such animals as swine and poultry, to control infections due to sensitive organisms and to promote growth. Supplements and mixed feeds containing these products can be stored for long periods of time without fear of appreciable change in bacitracin potency.

Example 1: A cation exchange resin is prepared in the following manner: A mixture of inhibitor-free styrene together with 2% divinylbenzene and 1% benzoyl peroxide is added to three times its volume of water in a reaction vessel equipped with agitator and reflux condenser. A trace of magnesium carbonate is added to facilitate suspension and the mixture is vigorously agitated and heated at about 90°C. until polymerization is complete. The resulting bead polymer is separated, washed and sulfonated by heating at 100°C. with an equal volume of concentrated sulfuric acid in the presence of 1% silver sulfate for 10 hours. The sulfonated copolymer is washed, dried, and screened to a particle size of 20 to 50 mesh. The resin, having a total exchange capacity of about 5 meq. per g. (dry basis), is then converted to the sodium form by treatment with aqueous sodium hydroxide followed by a water wash. The product has a moisture content of about 90%.

Example 2: 10 gallons of unfiltered plant fermentation broth containing 50 units per ml. of bacitracin is adjusted to pH 4 with dilute sulfuric acid and 400 grams of the undried resin (moisture content about 90%) of Example 1 is added. After an hour's vigorous stirring at room temperature the slurry is filtered and the filtrate, which contains considerably color, is assayed and discarded. Adsorption yield is about 97% and the adsorbate has a potency of about 24,000 units per g. (dry basis), equivalent to about 3,600 units per ml. of the wet resin.

The wet resin is charged to a small adsorption column containing an 80 mesh screen as a resin support, and the antibiotic activity is eluted with 9 liters of a 2% aqueous sodium bicarbonate solution having a pH of about 8.1. The eluant is passed over the column once at a constant rate in six hours.

The column effluent, having a potency of about 200 bacitracin u./ml. and a pH of about 7.5, is extracted batchwise with 3 one-third volumes of butanol. The butanol extracts are combined, washed with a half volume of distilled water to remove inorganic impurities, and extracted with 2 equal volumes of water adjusted to an equilibrium pH of about 2.5 with sulfuric acid. The water wash and the spent butanol are discarded. 2 gms. of activated decolorizing carbon are added to the two combined aqueous extracts and the pH is adjusted to about 5.1 with barium hydroxide. The resulting slurry is filtered, concentrated and freeze-dried to yield about 24 gms. of white bacitracin sulfate having a potency of 65 units per mg. and a moisture content of about 4%. Overall yield from broth to pure product is about 83%. A 500 u./ml. water solution of the product exhibits a 98.5% light transmission at 440 m $\mu$ .

Two grams quantities of the resin adsorbate above are stored at 104°C. for 24 hours. Duplicate initial and 24-hour samples are assayed and bacitracin activity retention under the test conditions is found to be 85%. A growth-stimulating feed for chicks is prepared by including the resin adsorbate above at a level of 3 grams bacitracin activity per ton of basal ration.

### Precipitation of Bacitracin Salts on Inert Support

Bacitracin salts are precipitated on an inert, insoluble, inorganic support such as diatomaceous earth in a process developed by C.H. Monroe and G.E. Ward (U.S. Patent 3,345,178; October 3, 1967; assigned to Dawe's Laboratories, Inc.).

Example: A bacitracin fermentation broth was adjusted to pH 4.3, filtered, and the filtrate was found to contain 92 units of bacitracin per ml. The filtered broth was concentrated in vacuo at 45°C. to obtain a solution which assayed 342 units per ml., and which contained 10.3% dissolved solids. The concentrated solution was cooled to 20°C. and to 1,600 ml. of the concentrate were added, with good mixing, 16 grams of Hyflo filter aid (a diatomaceous earth) and 400 grams of sodium chloride supplied in 50-gram increments, thereby forming a saturated solution. A precipitate formed, consisting principally of bacitracin deposited on the particles of Hyflo filter aid. The precipitate was separated by filtration and dried. The dry weight was 27.8 grams and the precipitate assayed 36.4% bacitracin. The dry product was free-flowing and was very suitable for incorporation in animal feeds and animal feed supplements. The bacitracin recovery efficiency, from concentrate to final product, was 77.7%.

## OTHER BACILLUS STRAINS

### Growth Factors from Specific Strains of *Bacillus Subtilis*

It has now been found that certain strains of the species *Bacillus subtilis* have the ability to biosynthesize nutritional factors useful for stimulating the growth of chicks and other animals. Thus cultures of the microorganisms, or concentrated preparations thereof, when fed to animals give growth responses which cannot be attributed to any of the known growth factors or to antibiotics which have been described previously and which have been recognized to be nutritionally active. In the case of one strain the growth stimulating effect is due (at least in part) to a new antibiotic, named aterrimin.



## Antibiotics as Anabolic Stimulators

The organisms which are used by J.C. Lewis, K. Ijichi, P.A. Thompson and J.A. Garibaldi; (U.S. Patent 2,942,977; June 28, 1960; assigned to the U.S. Secretary of Agriculture) are hitherto unknown strains of B. subtilis which were isolated from soil. Cultures of these organisms have been deposited in the Stock Culture Collection of the Northern Regional Research Laboratory, Peoria, Illinois, as Nos. NRRL B-1466, NRRL B-1471 and NRRL B-1474.

Example 1: A sterile medium was prepared containing the following ingredients dissolved in water.

| <u>Material</u>      | <u>Concentration<br/>g./liter</u> |
|----------------------|-----------------------------------|
| Beet molasses        | 85                                |
| Diammonium phosphate | 8.5                               |
| Diammonium citrate   | 10.0                              |
| Potassium sulphate   | 2.0                               |

In addition to the above ingredients, metal chloride salts were present in amounts to furnish the following concentration of metallic ions.

| <u>Ion</u> | <u>Concentration<br/>mg./liter</u> |
|------------|------------------------------------|
| Mg         | 50                                 |
| Ca         | 20                                 |
| Mn         | 50                                 |
| Fe         | 5                                  |
| Zn         | 5                                  |
| Co         | 2                                  |

The pH of the medium was adjusted to 7 by addition of ammonium hydroxide. Ten liters of the above medium contained in a fermenter similar to that disclosed by Humfield et al (U.S. Patent No. 2,542,031; February 20, 1951), was inoculated with 400 ml. of a shake culture of B. subtilis var. aterrimus NRRL B-1471. This shake culture had been prepared by inoculating 400 ml. of the beet molasses medium with a slant culture of the organism and then incubating 22 hours at 35°C. on a shaking machine.

The incubated medium was fermented at 35°C. employing constant agitation and forcing air into the medium at the rate of approximately 10 liters of air per minute. The pH of the culture was maintained at 6.3 to 7.0 by addition of ammonium hydroxide as needed. The growth of the organism was measured turbidimetrically in arbitrary units proportional to the optical density of the culture. At maximum growth (9 hours), the amount of cellular material on a dry basis was 28% of the sugar present.

The entire culture was vacuum concentrated at 50°C. and 30 inches Hg vacuum to about one-fifth the original volume. Into this concentrate was stirred an amount of corn syrup solids (500 grams), approximately equal to the weight of solids in the concentrate. The resulting mixture was poured in a shallow layer in a pan and frozen. The pan was then placed in a vacuum drier and dried for 24 hours at 30 inches vacuum without application

of heat, then for 24 hours under the same vacuum at 38°C. The culture was thus reduced to a dry, concentrated product which could be readily ground and mixed with feeds. The dried powder showed growth stimulating properties.

Example 2: The organism NRRL B-1471 was grown under aerated agitated submerged conditions on a liquid media as described above.

A. The resulting culture was acidified to pH 2.5 by the addition of concentrated hydrochloric acid. The precipitate including bacterial cells was isolated by centrifuging.

B. The precipitate from step A was blended with distilled water to give a volume of 615 ml. and the pH of the mixture adjusted to 2.5 by addition of hydrochloric acid; to this material was added 650 ml. of normal butyl alcohol and the mixture after thorough agitation was centrifuged to isolate the desired butanol extract from the aqueous phase. The aqueous phase was reextracted three times using 600 ml. of butanol in each operation. In these extractions additional water was added as necessary so that the volume of aqueous liquid and butanol would be about the same. Also the pH was maintained at pH 2.5 by addition of hydrochloric acid as needed. The four butanol extracts were then combined.

C. To the combined butanol extract from step B was added 200 g. sodium chloride and 5 ml. distilled water; the pH was adjusted to 5.0 by addition of sodium hydroxide. After stirring the mixture for 2 hours at room temperature it was filtered to remove undesired insoluble material and after allowing the filtrate to stand, the butanol phase was separated from the aqueous phase containing soluble impurities. The butanol phase was then evaporated under vacuum at room temperature to dryness.

D. The dry residue from step C was extracted 5 times with a total of 150 ml. of n-butanol leaving a residue of salt and insoluble brown material. The butanol extract, being a concentrated form of the growth-stimulating factor, was then made up to a volume of 160 ml. by addition of butanol. For tests to be described below the butanol extract was labeled VII D-1.

Example 3: Feeding tests were carried out using the butanol extract (product VII D-1) as a supplement to a basal ration for chicks. The tests were conducted on groups of 20 commercial hatchery New Hampshire chicks. For comparative purposes some of the lots of chicks were fed the basal ration by itself, this ration supplemented penicillin, and this ration supplemented with the dried, combined residues from the butanol extractions.

The materials used and the results obtained are tabulated on the following page.



| Feed supplement  |         | Average weight of chicks in grams or percentage of control at— |          | Feed required per gram of gain expressed in grams control at— |          |
|--|---------|--|----------|---|----------|
|  |         | 4 weeks  | 10 weeks | 4 weeks   | 10 weeks |
| No supplement (control)                                | g.      | 271  | 1,168    | 2.80  | 3.43     |
| Procaine penicillin G, 10 ppm                          | percent | 122  | 107      | 73  | 78       |
| Dried combined residues from butanol extractions, 0.4% | percent | 111  | 106      | 86  | 88       |
| Dried combined residues from butanol extractions, 0.2% | percent | 107  | 99       | 78  | 79       |
| Butanol extract (Prod. VII D-1) 0.1%                   | percent | 122  | 105      | 73  | 89       |
| Butanol extract (Prod. VII D-1) 0.033%                 | percent | 111  | 105      | 86  | 89       |

Inasmuch as 0.4% of the dried residue from the butanol extractions corresponded to 74 ml. of the original liquid whole culture per 100 g. of ration and 0.1% of the liquid butanol extract corresponded to 6.2 ml. of liquid whole culture per 100 g. of ration it is clear that approximately 90% of the chick-growth promoting activity appeared in the butanol extract as compared with the extraction residue.

#### Growth Promotor from *Bacillus Licheniformis*

W.N. McCutchan (U.S. Patent 3,261,688; July 19, 1966; assigned to Commercial Solvent Corporation) found that concentrated preparations from the culture of *Bacillus licheniformis* give a growth response to animals which cannot be attributed to bacitracin. Although it is presently not known whether the growth stimulating properties of the bacitracin-free elaboration product of the microorganism are due to constituents of an antibiotic or a vitamin character, the fact remains that important growth responses are obtained from their use. The microorganism *Bacillus licheniformis* is cultured on an aqueous nutrient medium, preferably under aerobic, submerged conditions.

Example 1: An aqueous fermentation medium containing the following proportions of ingredients was prepared.

|                   | Percent |
|-------------------|---------|
| Soybean meal      | 8       |
| Starch            | 2       |
| CaCO <sub>3</sub> | 0.25    |
| Lard oil          | 0.234   |

A 50-liter portion of the above medium was inoculated with 1,000 ml. of a shake flask culture of *Bacillus licheniformis*. The thus inoculated medium was fermented at 37°C. for 24 hours during which period air was forced into the medium at approximately 20 liters per minute. At the end of the 24-hour period, fermentation was halted and the fermentation medium was centrifuged to separate solid and liquid components. The aqueous component,

## Antibiotics as Anabolic Stimulators

rich in bacitracin, was removed from the fermentation medium by decantation and the solid material was dried by spray drying to give a solid product suitable for incorporation in animal feeds as growth stimulants.

Example 2: To demonstrate the effectiveness of the new growth-promoting agents, lambs were fed a complete feed containing 0.001% of the dried material of Example 1 for 28 days. Results are shown below.

| Treatment                                 | Number of Lambs | Average Daily Gain in Pounds | Pounds Average Daily Feed Consumed | Pounds Feed per Pound Gained |
|---|-----------------|------------------------------|------------------------------------|------------------------------|
| Control.....                              | 20              | 0.27                         | 2.73                               | 10.28                        |
| Control + Growth Stimulator (0.001%)..... | 20              | 0.43                         | 3.26                               | 7.61                         |

Example 3: Tests for 7 days on chicks showed:

| Treatment                                | Number of Chicks | Average Seven-Day Gain in Grams | Grams Feed per Grams Gained |
|--|------------------|---------------------------------|-----------------------------|
| Control.....                             | 100              | 68.5                            | 1.60                        |
| Control + Growth Stimulator (0.25%)..... | 100              | 71.1                            | 1.53                        |

### Growth Factors from *Bacillus Subtilis* and *Bacillus Natto*

T. Ukita, M. Nakai, Z. Minami, T. Yamazaki, and K. Ootaka (U.S. Patent 3,455,696; July 15, 1969; assigned to Nagase & Company, Ltd., Japan) have developed a process for producing an animal feed supplement wherein a microorganism selected from the group consisting of *Bacillus subtilis* and *Bacillus natto* is cultivated in a culture medium, the improvement comprising the steps of stopping the cultivation at a point between the beginning and middle of the logarithmic growth phase of the microorganism, and heating the resulting culture at a pH of 4.0 to 8.0 and at a temperature of 50° to 80°C. for 1 to 3 hours.

A culture medium (1,000 liters) having a pH of 7.0 containing 5% alkali extract of defatted soybeans, 4% starch, 2% lactose, 1% corn steep liquor and 1% (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> was charged in a cultivation tank (main tank) having a capacity of 2,000 liters.

Example: A *Bacillus subtilis* N<sup>+</sup> strain was cultivated at 37°C. under agitation and aeration. 50 liters of the culture broth were taken out at the second hour when the logarithmic growth phase sets in; the fourth hour which was the middle of the logarithmic growth phase; the sixth hour which was the latter part of the logarithmic growth phase; the tenth hour when the stationary phase sets in and the sixteenth hour, and were heated at 60°C. for 2 hours. The heat treated broth was absorbed in 50 kg. of bran and air dried at 45°C. and crushed to be an animal feed supplement. When Rockhorn F<sub>1</sub> (♂) chicks one day old were raised for tests for 8 weeks with basal feed, it was found that the effect of the increase



of the percentage of weight gain was remarkable with feed supplements obtained from the culture broth taken at or before the middle of the logarithmic growth phase; but that there was an action of inhibiting the growth of animals with those obtained from the culture broth taken thereafter. In this case, the basal feed used after the 5th week was of a productive energy of 1,200 cal./lb. and of 2% crude protein content. The addition of the feed supplement was 0.5% by weight based on the basal feed. The results are shown below.

EFFECTS OF RAISING WITH SUPPLEMENTS PREPARED AT DIFFERENT CULTURE AGES

| Pen No. | Supplements                  | Weight gain (percent) |        |        |        | Feed conversion |        |        |        |
|---------|------------------------------|-----------------------|--------|--------|--------|-----------------|--------|--------|--------|
|         |                              | 1 week                | 3 week | 6 week | 8 week | 1 week          | 3 week | 6 week | 8 week |
| 1       | 2nd hour's                   | 102.0                 | 105.1  | 106.0  | 107.0  | 1.59            | 1.89   | 2.46   | 2.82   |
| 2       | 4th hour's                   | 103.0                 | 105.3  | 106.2  | 106.8  | 1.59            | 1.81   | 2.40   | 2.72   |
| 3       | 6th hour's                   | 97.0                  | 102.0  | 101.5  | 101.8  | 1.57            | 1.83   | 2.42   | 2.79   |
| 4       | 10th hour's                  | 95.0                  | 95.3   | 94.6   | 97.1   | 1.61            | 1.84   | 2.45   | 2.80   |
| 5       | 16th hour's                  | 94.7                  | 95.5   | 96.5   | 99.3   | 1.58            | 1.82   | 2.38   | 2.75   |
| 6       | Control (without supplement) | 100                   | 100    | 100    | 100    | 1.66            | 1.89   | 2.59   | 2.98   |

Note.—100 chicks per pen were tested. Feed conversion: Represented by feed intake/weight gain.

## Insect Resistant Animal Feeds

A method for preparing insect-resistant animal feeds and for rendering area frequented by animals resistant to insect infestation has been developed by C.H. Koonz and R.A. Greenberg (U.S. Patent 3,285,748; November 15, 1966; assigned to Swift and Company). The feed composition comprises nutrient material for the animal and an insecticidal amount of the fermentation liquor obtained from the sporulation and proliferation of Bacillus thuringiensis Berliner microorganisms.

By "fermentation liquor" or "fermentation mash" is meant the nutrient medium remaining following the fermentation procedure which comprises the Bacillus thuringiensis Berliner cells, both vegetative and spore, metabolic products inherent in growth and/or sporulation, and unused components of the original medium. The solids content of the liquor comprises about 1% by weight, and these solids can be isolated by vacuum evaporation or other drying techniques. It is possible to prepare a solid product containing any desired amount of water by drying to a predetermined moisture level. The fermentation liquor in which the spores are proliferated can be admixed to the feed directly or the ferment can first be concentrated to provide a more concentrated insecticidal material. The type of feed treated can be any of that class of feeds generally characterized as dry feed, such as those containing leguminous plants, and seeds and the grain-producing grasses and cereals. Wet feeds, including canned pet foods, can also be treated with the spore-containing product.

Only a small insecticidally effective amount of a spore-containing composition need be present in the feed to obtain adequate insect control. Usually about 0.01 to 5% of the solids based on the weight of the feed is used. A significant decrease in the fly population in areas frequented by animals ingesting the feeds of this process has been noted with feeds that contain around 2% weight of Bacillus thuringiensis Berliner fermentation liquor.

Example: A medium formulation for proliferation of the spores is shown on the following page.

## Antibiotics as Anabolic Stimulators

### Component A:

|                   |     |     |
|-------------------|-----|-----|
| Cerelose          | 5   | g.  |
| Corn steep liquor | 4   | g.  |
| Ammonium sulfate  | 4   | g.  |
| Sodium hydroxide  | 0.2 | g.  |
| Water             | 900 | ml. |

### Component B:

|                      |     |     |
|----------------------|-----|-----|
| $K_2HPO_4$           | 1   | g.  |
| $MnSO_4 \cdot H_2O$  | 150 | mg. |
| $MgSO_4 \cdot 7H_2O$ | 1   | g.  |
| $ZnSO_4$             | 1   | mg. |
| $CuSO_4$             | 1   | mg. |
| $CaCl_2$             | 1   | mg. |
| $FeSO_4$             | 1   | mg. |
| Water                | 100 | ml. |

Each of the components A and B is autoclaved at 15 lbs. for 15 minutes and the 2 components are aseptically combined. The medium is inoculated with a loopful of Bacillus thuringiensis Berliner spores and the spores are proliferated in the medium for 18 to 24 hours at 90° to 95°F., with aeration and agitation. This material can then be used to inoculate a larger batch at a ratio of about 1 to 100 if desired. At the end of this time, 200 parts of the fermentation liquor, including the spores, vegetative cells, and the liquid, is mixed with 100 parts of pulverized soybean hulls. The mixture is dried by evaporation and the dried product is milled to reduce the lumps and the dried soybean hull material is employed as a component in animal feed compositions. This soybean hull material has been employed in animal feeds at a level of about 2.5% based on the weight of the feed.

A corn meal ration was prepared by incorporating in corn meal Bacillus thuringiensis Berliner fermentation liquor (41.2% wet weight) and the corn meal containing the insecticidal agent was employed in a feed containing 56.85% corn meal.

## STABILIZING TETRACYCLINE FEEDSTUFFS

### 7-Chloro-4-Epitetracycline

The use of the dried chlortetracycline-containing fermentation harvest mash solids as an animal or poultry feed supplement has from the beginning presented very vexing problems particularly the loss of potency of the antibiotic in the feed or feed supplement upon prolonged storage.

S. D. Upham and I. Klothen (U.S. Patent 3,023,105; February 27, 1962; assigned to American Cyanamid Company) found that the biologically inactive antibiotic 7-chloro-4-epitetracycline has the remarkable ability to stabilize the harvest mash solids to a considerable degree against loss in antibiotic potency over extended periods of time. It is believed that the epimer is gradually converted to the biologically active chlortetracycline, thus offsetting the loss of chlortetracycline potency which occurs by its possible conversion to the inactive



## Antibiotics as Anabolic Stimulators

epimeric form or possibly by its destruction to biologically inactive breakdown products. A commercial poultry feed containing 20% crude protein, 4% crude fat and 5% crude fiber was used in the following experiments.

Example: Chlortetracycline hydrochloride crystals in the amounts specified below were used in above poultry feed to determine antibiotic potencies after storage for varying periods of time. 7-chloro-4-epitetracycline in the amounts specified below was added to certain of the batches to prevent loss in antibiotic potency. The results obtained are shown in the table below.

| Composition   | Percent moisture | Average initial assay (microbiological) | Antibiotic recovery after storage as percent of initial assay |        |        |        |        |        |
|---|------------------|---|---|--------|--------|--------|--------|--------|
|   |                  |   | 23° C.  |        | 37° C. |        | 56° C. |        |
|   |                  |   | 1 wk.   | 4 wks. | 1 wk.  | 4 wks. | 1 wk.  | 4 wks. |
| Poultry feed CTC.HCl crystals, 200 grams/ton.....                                       | 13.0             | 207.0                                   | 91.5  | 81.0   | 81.5   | 59.0   | 36.0   | 6.4    |
| Do.....   | 16.6             | 198.0                                   | 95.0  | 76.5   | 67.5   | 34.0   | 9.6    | 2.1    |
| Poultry feed with blended crystals (200 gms./ton CTC.HCl, 200 gms./ton CTC epimer)..... | 12.8             | 199.0                                   | 110.0   | 111.0  | 98.5   | 100.5  | 72.5   | 60.5   |
| Poultry feed with blended crystals (200 gms./ton CTC.HCl, 200 gms./ton CTC epimer)..... | 16.7             | 187.0                                   | 103.0   | 123.0  | 100.0  | 80.5   | 58.0   | 35     |

### Maintaining Feed Supplement pH at 8.5 to 11.5

I. Klothen (U.S. Patent 3,019,109; January 30, 1962; assigned to American Cyanamid Company) found that a feed supplement could be stabilized against loss of antibiotic potency (1) by the addition of calcium hydroxide to an animal feed supplement, and which preferably is in the form of dried harvest mash solids with or without other feed materials and containing from 1 to 50 grams of chlortetracycline per pound, so as to raise or maintain the pH of the supplement to a pH of between about 7 and 12, and preferably 8.5 to 11.5, and (2) thereafter subjecting the so-treated supplement to a pelleting or agglomerating operation so as to produce pellets or crumbles having a particle size of the order of from 10 to 60 mesh.

A feed supplement sold commercially under the trademark Aurofac-10 chlortetracycline containing approximately 1 to 50 grams chlortetracycline per pound and consisting essentially of the following ingredients was prepared:

|                                     | <u>Percent</u> |
|-------------------------------------|----------------|
| Chlortetracycline fermentation cake | 53             |
| Coconut oil methylester             | 1.5            |
| Solvent extract soybean feed        | 45.5           |

Aurofac-10 chlortetracycline was used in a poultry feed containing 20% crude protein, 4% crude fat and 5% crude fiber, in a series of experiments with and without the addition of calcium hydroxide to determine losses in antibiotic potencies in the poultry feed after storage under accelerated conditions at 56°C. for two days. After the addition of calcium hydroxide to the feed supplement it was pelleted into 3/16 inch pellets and the so-treated feed

## Antibiotics as Anabolic Stimulators

supplement was then sized through a U.S. standard sieve No. 20 and retained on a U.S. standard sieve No. 30. The results obtained are shown in the following table.

| Composition  | Percent Moisture | Average Initial Assay (Microbiological) | Antibiotic Recovery After Storage as Percent of Initial Assay 56° C.—2 Days |
|--|------------------|---|---|
| Aurofac-10 in Poultry Feed.....  | 17.6             | 204.5                                   | 64.5  |
| Aurofac-10 Crumbles in Poultry Feed.....   | 17.6             | 185.5                                   | 66.0  |
| Aurofac-10 in Poultry Feed with 5% Ca(OH) <sub>2</sub> .....   | 17.3             | 181.5                                   | 71.0  |
| Aurofac-10 Crumbles in Poultry Feed with 5% Ca(OH) <sub>2</sub> .....                                    | 17.4             | 167.5                                   | 89.5  |
| Aurofac-10 <sup>1</sup> in Poultry Feed CTC/calcium caseinate with 5% Ca(OH) <sub>2</sub> .....          | 17.3             | 184.5                                   | 78.5  |
| Aurofac-10 <sup>1</sup> Crumbles in Poultry Feed CTC/calcium caseinate with 5% Ca(OH) <sub>2</sub> ..... | 17.5             | 185.0                                   | 82.5  |

<sup>1</sup> Chlortetracycline/calcium caseinate complex was used in lieu of chlortetracycline fermentation cake. This material has a potency of 60% and was used at 4.3% of the total blend. The remaining diluent was supplied from soybean feed.

### pH Adjustments to Improve Stability

In commercial practice, a typical procedure for obtaining the dried harvest mash antibiotic-containing solids is carried out by harvesting the fermentation mash. S.A. Muller (U.S. Patent 3,157,512; November 17, 1964; assigned to American Cyanamid Company) found that instead of harvesting and drying the fermentation mash at the weakly alkaline pH of about 6 to 8, if the mash is first acidified to a pH of about 0.1 to 3 and then readjusted upward to an alkaline pH of around 8.5 to 13, the dried mash solids are stabilized against loss of antibiotic potency upon storage for prolonged periods of time.

When the stable feed supplement is blended with conventional poultry feed materials, it has been found that there is a distinct loss in antibiotic potency of the finished feed upon storage for prolonged periods of time, which can be prevented by the addition of mild caustic, preferably calcium hydroxide, to the finished feed in a quantity sufficient to raise and maintain the pH to between 6 and 12 and preferably 8.5 to 11.5.

**Example 1:** A. Conventional preparation of feed supplements: a 1-liter portion of harvest mash obtained as a result of *Streptomyces aureofaciens* strain A-377 fermentation for 120 hours at 28°C. was adjusted to pH 7.2 with 15 N NaOH solution. 20 g. of diatomaceous earth filter-aid and 2 g. of magnesium silicate were added. The slurry was stirred for 10 minutes, then filtered. The filter cake was air dried overnight to a volatiles content of 8.5%. This product assayed 92 mcg. of chlortetracycline per milligram of solids by microbiological assay. After standing in a tightly-capped jar at 56°C. for 7 days, the product assayed 65 mcg./mg., a loss of 26% of the original activity.

B. This Process — Another 1-liter portion of this harvest mash was adjusted to pH 1.7 by the addition of concentrated HCl. A 20 g. quantity of diatomaceous earth filter-aid and 2 grams of magnesium silicate were added and the slurry stirred. After 5 minutes of stirring, 15 N sodium hydroxide solution was used to adjust the pH to 8.4. The slurry was filtered and the filter cake dried to a volatiles content of 7.8%. This cake assayed 90.5 mcg. of chlortetracycline per milligram of solids. After being kept 7 days in a tightly-capped jar at 56°C., this product assayed 89 mcg./mg.; a loss of only 1.7% of the original activity



as compared to the 26% loss in the control.

Example 2: Feed supplements were prepared as in the example using both the conventional procedure and that of this process, to obtain chlortetracycline-containing harvest mash solids having moisture contents of about 17%. These two types of solids were assayed for chlortetracycline content.

| Feed Supplement Type | Treatment       | Initial Assay for CTC, mcg./g. | Loss in Potency at 56° C. for 7 days, percent |
|----------------------|-----------------|--------------------------------|---|
| Conventional.....    | pH 7.2.....     | 444                            | 22.6  |
| New Process.....     | pH 1.7-pH 8.4.. | 479                            | 3.5   |

Chick feeds were prepared using the following proportions:

|                               |          |
|-------------------------------|----------|
| (a) Standard chick feed       | 100.0 g. |
| Ca(OH) <sub>2</sub>           | 2.0 g.   |
| Conventional procedure solids | 0.5 g.   |
| (b) Standard chick feed       | 100.0 g. |
| Ca(OH) <sub>2</sub>           | 2.0 g.   |
| This Process Solids           | 0.5 g.   |

and thus maintaining, by means of the Ca(OH)<sub>2</sub> a pH of 9.0 in the final chick feed blends.  
Chlotetracyline assay:

| Feed type:          | Loss in potency at 56°C. for 7 days |
|---------------------|-------------------------------------|
| Conventional        | 26 percent                          |
| This Process Solids | 2 percent                           |

#### Addition of Aluminum Salts Prior to Harvesting Mash

In another process, A. Abbey, R.B. Fortenbaugh, and I. Klothen (U.S. Patent 3,427,166; February 11, 1969; assigned to American Cyanamid Company) found that if aluminum ion is added to the fermentation mash prior to harvest and the harvesting is thereafter carried out in a convention manner, the resulting dried harvest mash solids containing the antibiotic are stable against losses in antibiotic potency for extended periods of time whether or not it is in the usual form of a dried cake or whether it is blended with conventional poultry or animal feed materials.

The chlortetracycline test mashers were prepared by withdrawing representative 100 ml. samples from the fermentation vats upon completion of fermentation and prior to harvesting. These 10 ml. samples were placed in beakers and the desired amount of aluminum salt in 20 ml. of water was admixed therewith. The pH value of these mixtures was generally found to be between pH 3.2 to 3.5.

## Antibiotics as Anabolic Stimulators

A sufficient amount of sodium hydroxide solution was then added to each mixture to adjust the pH thereof to a value of from 7.0 to 7.2. Then with continued stirring, the mixtures were heated to from 90° to 100°C. and maintained at such temperature for about five minutes. The mixtures were cooled to about 25°C. and, where necessary, the pH readjusted to about 7.2. Approximately 1.5 grams of Celite No. 512 filter aid was introduced into the cool mixture and the entire mixture filtered through Whatman No. 1 filter paper previously coated with 1.5 grams of Celite No. 512. The filtrate was discarded and the filter cake transferred to a Petri dish and dried in a vacuum oven for 18 to 22 hours at about 48° to 55°C. and 25 inches of vacuum. The dried filter cake was then ground to a fine uniform powder, resuspended in water to original treatment volume (130 ml.) and approximately 13 ml. (1/10 original mash volume) from each treatment was placed in a number of test tubes.

Control samples for each treatment were simply capped and stored without the addition of feed thereto. However, to substantially identical samples from each treatment were added 10 grams of a commercially available poultry feed (a minimum of 20% crude protein, minimum crude fat 4% and maximum crude fiber 5%). All samples were stored at 56°C. for three days, then removed from storage and assayed microbiologically for chlortetracycline content.

The instant tests run at 56°C. in the presence of water with and without feed are accelerated tests designed to create severe conditions and produce results one would expect to obtain on prolonged storage (i.e., a year or more) under normal conditions.

| Preparation of Supplement – Filtered and Dried |  |                |            | Prep. of Samples for Tests—Filter Cake Resuspended in 130 ml. H <sub>2</sub> O, ml./test tube | Microbioassay Test Tube Contents, mcg. CTC/Test Tube—Plain and With Poultry Feed Added After 3 Days' Storage at 56° C |                                 |
|--|--|----------------|------------|---|---|---------------------------------|
| ml. Mash                                       | Additive   | Heat, °C./min. | Filter Aid |   | Plain, No Feed Added  | Feed Added, 10 g. per test tube |
| 100 ml. CTC                                    | None   | None           | Celite 512 | 13  | 70,000  | 23,500                          |
| 100 ml. CTC                                    | None, adj. to pH 3.2 with HCl                        | 90-100/5       | do         | 13  | 58,000  | 28,250                          |
| 100 ml. CTC                                    | None   | 90-100/5       | do         | 13  | 70,500  | 32,200                          |
| 100 ml. CTC                                    | 1.25% AlCl <sub>3</sub>                              | None           | do         | 13  | 49,150  | 40,250                          |
| 100 ml. CTC                                    | 1.25% AlCl <sub>3</sub>                              | 90-100/5       | do         | 13  | 45,250  | 42,700                          |
| 100 ml. CTC                                    | 1.25% AlCl <sub>3</sub>                              | 90-100/5       | do         | 13  | 49,750  | 48,250                          |
| 100 ml. CTC                                    | 1.25% AlCl <sub>3</sub>                              | 90-100/5       | do         | 13  | 55,250  | 49,500                          |
| 100 ml. CTC                                    | 1.6% Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> | 90-100/5       | do         | 13  | 56,000  | 50,750                          |

| Preparation of Supplement – Filtered and Dried |                         |                |                   | Prep. of Samples for Tests—Filter Cake Resuspended in H <sub>2</sub> O, ml./test tube | Microbioassay Test Tube Contents, mcg. CTC/Test Tube—Plain and With Poultry Feed Added After 3 Days' Storage at 56° C |                                 |
|--|-------------------------|----------------|-------------------|---|---|---------------------------------|
| ml. Mash                                       | Additive                | Heat, °C./min. | Filter Aid        |   | Plain, No Feed Added  | Feed Added, 10 g. per test tube |
| 100 ml. CTC                                    | None                    | 90-100/5       | 3% Celite 512     | 13  | 67,750  | 34,250                          |
| 100 ml. CTC                                    | 1.25% AlCl <sub>3</sub> | 90-100/5       | do                | 13  | 60,250  | 51,500                          |
| 100 ml. CTC                                    | 1.25% AlCl <sub>3</sub> | 90-100/5       | 2.4% Dicalite 436 | 13  | 64,500  | 56,000                          |

### Heat Treatment Prior to Harvesting Mash

The fermentation mash solids can be stabilized against loss of antibiotic potency by heating for at least three minutes prior to harvesting, according to this process of I. Klothen, R. B. Fortenbaugh, and A. Abbey (U.S. Patent 3,434,844; March 25, 1969; assigned to American Cyanamid Company).



## Antibiotics as Anabolic Stimulators

Example: 100 milliliters of liquid mash which had been prepared in accordance with conventional fermentation techniques were withdrawn from the fermentation vat just prior to harvesting. These samples were placed in containers, 1.5 grams of filter aid was added and the mixture placed in an autoclave, and heat treated at 121°C. for 3 minutes. They were then removed from the autoclave and permitted to cool and 10 grams of liquid mash was weighed into test tubes and assayed in a chlortetracycline microbioassay test tube (contents mcg./test tube) 3 days after storage at 56°C.

| Gm. Mash                | Heat      | Filter Aid               | Plain, No<br>Feed Added | Feed Added<br>10 g. per<br>Test Tube |
|-------------------------|-----------|--------------------------|-------------------------|--------------------------------------|
| 10 gm. liquid mash..... | None..... | 1.5 g. Celite No. 512... | 82,250                  | 44,250                               |
| Do.....                 | (1)       | do.....                  | 93,250                  | 49,750                               |

<sup>1</sup> Autoclaved for 3 min. at 121° C.

It has also been found that if the dried feed supplement is crushed to a suitable particle size additional stability is imparted to the resulting product. A supplement having an average particle size of plus 325 mesh or approximately 44 microns reduces loss of antibiotic potency by approximately 20% over similar material having a particle size of minus 325 mesh or approximately 20 microns. The preferred particle size is 44 to 600 microns (30 U.S. screen).

## TETRACYCLINE IN FEED SUPPLEMENTS

### Forms of Calcium and Phosphorus in Tetracycline Diets

It has been found in the past that when a small amount of a broad spectrum antibiotic of the tetracycline type is added to the feed of animals, improved health and therefore growth results. This creates a problem because in the past the standard sources for calcium and phosphorus, calcium carbonate and calcium hydrogen phosphate or bone meal, markedly lower the blood levels of the tetracycline antibiotics.

C.N. Huhtanen and W.L. Williams (U.S. Patent 2,962,378; November 29, 1960; assigned to American Cyanamid Company) found that satisfactory blood levels of tetracycline antibiotic could be obtained with a diet having a full normal amount of calcium and phosphorus. This is based on the surprising discovery that the adverse effects of a normal calcium diet on tetracycline antibiotic blood levels are not due solely to the presence of calcium but rather to the form in which the calcium is present. Thus, the deleterious effects are noted when calcium carbonate is used, the standard source of calcium, and when the phosphorus source is a calcium phosphate, such as calcium hydrogen phosphate. With exactly the same total amount of calcium and phosphorus, much higher blood levels are obtained, comparable to those obtained by a low calcium diet or by the addition of terephthalic acid, when calcium sulfate is used as a source of calcium and when the phosphorus source itself does not contain a compound of calcium.

Example: Groups of six 27 day old chicks were placed on a low calcium diet with various calcium and phosphorus supplements. There were used in each case two different kinds of

## Antibiotics as Anabolic Stimulators

tetracycline antibiotics, CTC and tetracycline (abbreviated TC). The results are shown below.

| Supplement to Low Calcium Diet  | Blood Levels  |              |
|---|---------------|--------------|
|   | CTC,<br>γ/ml. | TC,<br>γ/ml. |
| None.....   | 0.33          | 0.30         |
| CaCO <sub>3</sub> +CaHPO <sub>4</sub> ·2H <sub>2</sub> O.....             | 0.15          | 0.14         |
| CaSO <sub>4</sub> ·2H <sub>2</sub> O+H <sub>3</sub> PO <sub>4</sub> ..... | 0.30          | 0.23         |

### Antibiotic, Estrogen, Anthelmintic Combination

W.P. Johnson and R.F. Elliott (U.S. Patent 2,929,712; March 22, 1960; assigned to American Cyanamid Company) found that animals showed a significant increase in growth rate when fed on an additive containing a combination of tetracycline antibiotic, estrogen derivative, and an anthelmintic agent.

Eighty 650-pound feeder steers of uniform weight and conformation were placed in 16 pens with 5 head per pen. The various groups of steers were fed on a standard feed yard basal ration with some of the groups receiving chlortetracycline, some receiving stilbestrol, some receiving phenothiazine, and some receiving combinations of these additives. The quantity of chlortetracycline was about 75 mg. per steer per day, the quantity of stilbestrol about 10 mg. per steer per day, and the quantity of phenothiazine about 2 grams per steer per day. The results of this feeding program appear below. The rations and supplements utilized are as follows:

|               |                           |      |      |
|---------------|---------------------------|------|------|
| Basal ration: | Corn chop                 | 500  | lbs. |
|               | Quaker oats molasses meal | 300  | lbs. |
|               | Milo                      | 300  | lbs. |
|               | Bran                      | 140  | lbs. |
|               | Cottonseed hulls          | 200  | lbs. |
|               | Cottonseed meal           | 50   | lbs. |
|               | Linseed meal              | 50   | lbs. |
|               | Schumacher milo           | 300  | lbs. |
|               | Total                     | 1840 | lbs. |

|               |                                    |         |      |
|---------------|------------------------------------|---------|------|
| Supplement C: | Dicalcium phosphate                | 20.0    | lbs. |
|               | Sodium chloride                    | 25.0    | lbs. |
|               | Calcium carbonate                  | 25.0    | lbs. |
|               | Trace mineral mix                  | 5.0     | lbs. |
|               | Vitamin A (10,000 units per grams) | 25.0    | lbs. |
|               | Fortafeed 249C                     | 6.25    | lbs. |
|               | Soybean meal (Aurofac grade)       | 16.0    | lbs. |
|               | Alfalfa meal (17%)                 | 816.55  | lbs. |
|               | Citrus meal                        | 61.2    | lbs. |
| Total         |                                    | 1000.00 | lbs. |



## Antibiotics as Anabolic Stimulators

|               |                                   |         |      |
|---------------|-----------------------------------|---------|------|
| Supplement A: | Aurofac-10 <sup>1</sup>           | 137.0   | lbs. |
|               | Dicalcium phosphate               | 20.0    | lbs. |
|               | Sodium chloride                   | 25.0    | lbs. |
|               | Calcium carbonate                 | 25.0    | lbs. |
|               | Trace mineral mix <sup>2</sup>    | 5.0     | lbs. |
|               | Vitamin A (10,000 units per gram) | 25.0    | lbs. |
|               | Fortafeed 249C <sup>3</sup>       | 6.25    | lbs. |
|               | Alfalfa meal (17%)                | 756.75  | lbs. |
|               | Total                             | 1000.00 | lbs. |

<sup>1</sup>Aurofac-10 contains approximately 10 to 12 grams of chlortetracycline per pound.

<sup>2</sup>Trace Mineral Mix contains about 6% manganese as the oxide thereof, about 2% iron as the carbonate, about 0.2% copper as the hydroxide, about 0.12% iodine as potassium iodide, about 0.02% cobalt as the carbonate, and between about 26.5 and 31.8% calcium as the stearate.

<sup>3</sup>Fortafeed 249C is a vitamin mixture containing per pound 4 grams of pantothenic acid, 2 grams of riboflavin, 9 grams of niacin, 16 grams of choline chloride and 64 mg. of folic acid and soybean meal.

|               |                               |         |      |
|---------------|-------------------------------|---------|------|
| Supplement D: | Dicalcium phosphate           | 20.0    | lbs. |
|               | Sodium chloride               | 25.0    | lbs. |
|               | Calcium carbonate             | 25.0    | lbs. |
|               | Trace mineral mix             | 5.0     | lbs. |
|               | Vitamin A (10,000 units)      | 25.0    | lbs. |
|               | Fortafeed 249C                | 6.25    | lbs. |
|               | Soybean meal (Aurofac grade)  | 16.0    | lbs. |
|               | Citrus meal                   | 61.2    | lbs. |
|               | Alfalfa meal (17%)            | 795.75  | lbs. |
|               | Stilbestrol premix (2 g./lb.) | 20.8    | lbs. |
|               | Total                         | 1000.00 |      |

|               |                              |         |      |
|---------------|------------------------------|---------|------|
| Supplement P: | Dicalcium phosphate          | 20.0    | lbs. |
|               | Sodium chloride              | 25.0    | lbs. |
|               | Calcium carbonate            | 25.0    | lbs. |
|               | Trace mineral mix            | 5.0     | lbs. |
|               | Vitamin A (10,000 units)     | 25.0    | lbs. |
|               | Fortafeed 249C               | 6.25    | lbs. |
|               | Soybean meal (Aurofac grade) | 16.0    | lbs. |
|               | Citrus meal                  | 61.2    | lbs. |
|               | Alfalfa meal (17%)           | 806.55  | lbs. |
|               | Phenothiazine (402 g./lb.)   | 10.0    | lbs. |
|               | Total                        | 1000.00 | lbs. |

In each group of feedings the supplements were added to 1840 lbs. of basal ration, for a total of 2,000 lbs. ration. Controls in two groups, I and Ia, were fed on the basal ration, plus 160 lbs. of supplement C.

Groups II and IIa received about 75 mg. of chlortetracycline per steer per day with supplement A 6 lbs. and supplement C 154 lbs. Groups III and IIIa received about 10 mg. of

stilbestrol per steer per day with supplement D 24 lbs. and supplement C 136 lbs. Groups IV and IVa received about 2 g. of phenothiazine per steer per day with supplement P 40 lbs. and supplement C 120 lbs. Groups V and Va received about 75 mg. of chlortetracycline per steer per day plus about 10 mg. of stilbestrol per steer per day with supplement A 6 lbs., supplement D 24 lbs., and supplement C 130 lbs. Groups VI and VIa received about 75 mg. of chlortetracycline, plus about 2 g. of phenothiazine respectively per steer per day with supplement A 6 lbs., supplement P 40 lbs., and supplement C 114 lbs. Groups VII and VIIa received about 10 mg. of stilbestrol, plus about 2 g. of phenothiazine respectively per steer per day with supplement D 24 lbs., supplement P 40 lbs., and supplement C 96 lbs. Groups VIII and VIIIa received about 10 mg. of stilbestrol, about 2 g. of phenothiazine and about 75 mg. of chlortetracycline respectively per steer per day with supplement D 24 lbs., supplement P 40 lbs., supplement A 6 lbs., and supplement C 90 lbs.

Each of the eight paired groups of steers were weighed at the seventh day, the thirty-fifth day, the sixty-third day, the ninety-first day and the one hundred-nineteenth day. The table that follows illustrates the observations made between the first and one hundred-nineteenth days inclusive, and are representative of the observations made throughout the feeding program.

| Groups             | Average Weight In Pounds | Average Gain In Pounds | Cost Per Pound of Gain |
|--------------------|--------------------------|------------------------|------------------------|
| I and IA.....      | 1,052                    | 406                    | \$ .217                |
| II and IIA.....    | 1,087                    | 427                    | .206                   |
| III and IIIA.....  | 1,071                    | 439                    | .214                   |
| IV and IVA.....    | 1,014                    | 376                    | .232                   |
| V and VA.....      | 1,082                    | 435                    | .207                   |
| VI and VIA.....    | 1,054                    | 401                    | .227                   |
| VII and VIIA.....  | 1,070                    | 429                    | .210                   |
| VIII and IIIA..... | 1,124                    | 473                    | .195                   |

It is particularly significant to note that the addition of the combination of chlortetracycline, diethylstilbestrol and phenothiazine substantially enhances the growth rate of the steers in the above feeding experiment.

#### Antibiotic Plus Stilbene Derivative

H.G. Luther and W.M. Reynolds (U.S. Patent 2,951,760; September 6, 1960; assigned to Chas. Pfizer and Company Inc.) found that feedstuffs containing both the antibiotic and estrogen produced better growth results than those containing either additive alone.

Example 1: A cattle experiment was conducted with 64 Hereford steers divided into four lots of 16, average initial weight of 750 lbs., which were fed in groups the following supplements for a 98-day fattening period.

- A. Basal ration (control)
- B. Basal+OTC, 160 mg. per head per day (5.0 mg./lb. feed)
- C. Basal+DES, 10 mg. per head per day (0.31 mg./lb. feed)
- D. Basal+DES, 10 mg.+OTC, 160 mg. per head per day



## Antibiotics as Anabolic Stimulators

The basal ration used was a complete feed of the following composition:

|             |                        |     |         |
|-------------|------------------------|-----|---------|
| Ingredient: | Cottonseed hulls       | 22  | percent |
|             | Ground corn cobs       | 8   | percent |
|             | Ground yellow corn     | 22  | percent |
|             | Molasses               | 10  | percent |
|             | Barley                 | 25  | percent |
|             | Cottonseed meal        | 9   | percent |
|             | Dicalcium phosphate    | 2   | percent |
|             | Limestone              | 1   | percent |
|             | Trace mineralized salt | 1.0 | percent |
|             |                        | 100 |         |

In addition, vitamin A was supplied at the rate of 200,000 I.U. per 100 lbs. The results of the trial are set forth in the following table.

| Gain                             | A<br>Con-<br>trol | B<br>OTC* | C<br>DES* | D<br>OTC+DES |
|----------------------------------|-------------------|-----------|-----------|--------------|
| Average Daily Gain.....          | 2.37              | 2.45      | 2.61      | 2.97         |
| Growth Index.....                | 100               | 103       | 110       | 125          |
| Feed Efficiency:                 |                   |           |           |              |
| Lb. Feed/Lb. Gain.....           | 13.05             | 12.94     | 11.80     | 10.80        |
| Feed Efficiency Index.....       | 100               | 101       | 111       | 121          |
| Dollar Savings Over Control..... |                   | \$0.46    | \$5.12    | \$8.18       |

\*OTC=oxytetracycline; DES=diethylstilbestrol.

Example 2: A further experiment was conducted with 68 Hereford steer calves divided equally into four groups of 17 animals. The animals were fed in feedlot for a 168-day test period and received the following supplements to the basal ration:

- A. Basal ration (control)
- B. Basal+OTC, 10 mg. per 100 lbs. live weight per day  
(2 mg./lb. feed)
- C. Basal+DES, 10 mg. per head per day (0.28 mg./lb. feed)
- D. Basal+DES, 10 mg. per head+OTC, 10 mg. per 100 lbs.  
live weight per day

The following average daily ration was consumed:

|             |                      |             |
|-------------|----------------------|-------------|
| Ingredient: | Cottonseed meal      | 1.44        |
|             | Milo grain           | 7.9-8.57    |
|             | Alfalfa hay, chopped | 2.78-3.02   |
|             | Hegari silage        | 19.92-21.49 |

The table on the following page sets forth the final results of this experiment.

## Antibiotics as Anabolic Stimulators

| Gain                             | A<br>Con-<br>trol | B<br>OTC* | C<br>DES* | D<br>OTC+DES |
|----------------------------------|-------------------|-----------|-----------|--------------|
| Average Daily Gain.....          | 2.1               | 2.00      | 2.18      | 2.41         |
| Growth Index.....                | 100               | 95        | 104       | 115          |
| Feed Efficiency:                 |                   |           |           |              |
| Lb. Feed/Lb. Gain.....           | 15.26             | 16.11     | 14.74     | 14.33        |
| Feed Efficiency Index.....       | 100               | 94        | 103       | 106          |
| Dollar Savings Over Control..... |                   |           |           |              |

\*OTC=oxytetracycline; DES=diethylstilbestrol.

### Tetracycline, Sulfonamide, Penicillin Combination

The process of M. J. Harvey (U. S. Patent 3,185,573; May 25, 1965; assigned to American Cyanamid Company) is concerned with a combination of a tetracycline antibiotic, a sulfonamide such as sulfamethazine or sulfaethoxypyridazine, and penicillin as an animal feed additive composition which significantly enhances the growth rate of animals, particularly early weaned pigs.

[Body weight gains (lbs.) of pigs infected with *Salmonella choleraesuis* var. *kunz*, and medicated with 100 gms./ton chlortetracycline, 100 gms./ton sulfamethazine, 49 gms./ton penicillin in the feed and pigs on non-medicated feeds when infected and non-infected]

| Group   | Treatment  | Original weight of separate pigs | Weight gain (lbs.)/pig/group/week post-infection |          |          |       | Final weight |
|---------|--|----------------------------------|--|----------|----------|-------|--------------|
|         |  |                                  | 1st week   | 2nd week | 3rd week | Total |              |
| 1       | Non-infected; non-medicated.....   | 47.5                             | 15.5   | 13.5     | 17.5     | 46.5  | 94.0         |
|         |  | 37.5                             | 10.5   | 10.0     | 7.0      | 27.5  | 65.0         |
|         |  | 40.0                             | 10.5   | 10.0     | 12.5     | 33.0  | 73.0         |
|         |  | 37.0                             | 5.5  | 8.0      | 11.0     | 24.5  | 61.5         |
|         |  | 35.5                             | 7.0  | 7.5      | 9.5      | 24.0  | 59.5         |
| Average |  |                                  |  |          |          |       |              |
| Total   |  |                                  | 9.8  | 9.8      | 11.5     | 31.1  |              |
|         |  | 197.5                            |  |          |          |       | 353.0        |
| 1A      | Non-infected; non-medicated.....   | 42.0                             | 10.0   | 10.0     | 11.0     | 31.0  | 73.0         |
|         |  | 43.0                             | 10.5   | 13.5     | 13.0     | 37.0  | 80.0         |
|         |  | 39.0                             | 12.0   | 11.0     | 13.5     | 36.5  | 75.5         |
|         |  | 38.0                             | 16.5   | 12.5     | 13.0     | 42.0  | 80.0         |
|         |  | 34.0                             | 9.0  | 10.5     | 10.5     | 30.0  | 64.0         |
| Average |  |                                  |  |          |          |       |              |
| Total   |  | 190.0                            | 11.6   | 11.5     | 12.2     | 35.3  | 372.5        |
| 2       | Infected, non-medicated.....   | 49.0                             | -5.5D  |          |          |       |              |
|         |  | 38.0                             |  |          |          |       |              |
|         |  | 40.0                             | 0  | 2.0      | 6.0      | 8.0   | 48.0         |
|         |  | 35.0                             | 0  | 6.0      | 6.5      | 12.5  | 47.5         |
|         |  | 36.5                             | -2.0   | 0.5      | 4.5      | 3.0   | 39.5         |
| Average |  |                                  |  |          |          |       |              |
| Total   |  | 198.5                            | -1.9   | 2.8      | 5.7      | 7.8   | 135.0        |
| 2A      | Infected; non-medicated.....   | 41.0                             | -1.0   | 1.0      | 0        | 0     | 41.0         |
|         |  | 41.0                             | -3.5D  |          |          |       |              |
|         |  | 39.0                             | -3.0   | 4.5      | 11.5     | 13.0  | 52.0         |
|         |  | 39.0                             | -4.0   | -8.0     | D        |       |              |
|         |  | 38.0                             | -1.0   | 1.0      | -4.0     | -4.0  | 34.0         |
| Average |  |                                  |  |          |          |       |              |
| Total   |  | 198.0                            | -2.5   | -0.13    | 2.5      | 3.0   | 127.0        |
| 3       | Infected, medicated feed, 100 gms./ton chlortetracycline, 100 gms./ton sulfamethazine, 49 gms./ton penicillin..... | 48.5                             | 3.5  | 11.0     | 13.0     | 27.5  | 76.0         |
|         |  | 38.5                             | 1.5D   |          |          |       |              |
|         |  | 38.0                             | 9.0  | 10.0     | 9.5      | 28.5  | 66.5         |
|         |  | 36.0                             | 7.0  | 11.0     | 11.0     | 29.0  | 65.0         |
|         |  | 36.5                             | 6.0  | 11.5     | 11.0     | 28.5  | 65.0         |
| Average |  |                                  |  |          |          |       |              |
| Total   |  | 197.5                            | 5.4  | 10.9     | 11.1     | 28.3  | 272.5        |
| 3A      | Infected, medicated feed, 100 gms./ton chlortetracycline, 100 gms./ton sulfamethazine, 50 gms./ton penicillin..... | 45.0                             | 4.5  | 14.0     | 13.5     | 32.0  | 71.0         |
|         |  | 41.0                             | 1.5  | 9.5      | 11.0     | 22.0  | 63.0         |
|         |  | 40.0                             | 6.0  | 11.0     | 11.0     | 28.0  | 68.0         |
|         |  | 39.0                             | 10.0   | 16.0     | 14.5     | 40.5  | 79.5         |
|         |  | 36.0                             | -1.0   | 4.5      | 13.5     | 17.0  | 53.0         |
| Average |  |                                  |  |          |          |       |              |
| Total   |  | 201.0                            | 4.2  | 11.0     | 12.7     | 27.9  | 340.5        |

D=Died between last indicated weigh day and following one.



## Antibiotics as Anabolic Stimulators

The table on the previous page shows the results of an experiment with 30 pigs of essentially equal weight divided into 6 equal groups. Infected and noninfected control groups were established. Two noninfected control groups were placed on a standard, starter, non-medicated pig diet. The second control groups were infected with the test organism, Salmonella choleraesuis var. kunzendorf, and placed on the same non-medicated, starter pig diet. The final test groups were infected with the same organism and were placed on the standard diet which had been medicated with 100 g./ton of chlortetracycline, 100 g./ton of sulfamethazine, and 49 g./ton of penicillin.

To establish the effect of introducing into the feed of weanling pigs a combination of chlortetracycline, sulfamethazine and penicillin at very low levels, the following experiment was undertaken. Thirty weanling pigs were divided into six equal groups and permitted to feed and drink ad libitum. A standard, starter pig diet to which was added the particular levels of chlortetracycline, sulfamethazine, and penicillin is set forth below.

|                                 |       |       |       |       |       |       |
|---------------------------------|-------|-------|-------|-------|-------|-------|
| Chlortetracycline, gm./ton..... | 0     | 1.25  | 10    | 20    | 30    | 40    |
| Sulfamethazine, gm./ton.....    | 0     | 1.25  | 10    | 20    | 30    | 40    |
| Penicillin, gm./ton.....        | 0     | 0.63  | 5     | 10    | 15    | 20    |
| Pigs per group—replotted.....   | 5     | 5     | 5     | 5     | 5     | 5     |
| Average weights:                |       |       |       |       |       |       |
| Initial.....                    | 32.5  | 33.7  | 34.3  | 32.6  | 31.0  | 36.2  |
| After 1st 14 days.....          | 46.6  | 47.0  | 52.4  | 48.3  | 46.1  | 55.1  |
| After 2nd 14 days.....          | 64.3  | 60.0  | 75.1  | 69.1  | 66.9  | 77.1  |
| After 3rd 14 days.....          | 82.5  | 85.5  | 96.9  | 92.0  | 87.8  | 100.0 |
| After 4th 14 days.....          | 100.0 | 102.6 | 118.1 | 114.6 | 107.7 | 121.5 |
| After 5th 14 days.....          | 125.1 | 124.0 | 144.9 | 142.7 | 135.0 | 149.1 |
| After 6th 14 days.....          | 153.6 | 149.2 | 172.6 | 170.5 | 161.8 | 179.5 |
| After 7th 14 days.....          | 176.5 | 167.2 | 193.4 | 194.4 | 184.6 | 201.5 |
| Average daily gain:             |       |       |       |       |       |       |
| After 1st 14 days.....          | 1.01  | 0.95  | 1.29  | 1.12  | 1.08  | 1.35  |
| After 2nd 14 days.....          | 1.26  | 1.36  | 1.63  | 1.49  | 1.49  | 1.57  |
| After 3rd 14 days.....          | 1.30  | 1.39  | 1.66  | 1.64  | 1.60  | 1.64  |
| After 4th 14 days.....          | 1.25  | 1.22  | 1.52  | 1.62  | 1.42  | 1.64  |
| After 5th 14 days.....          | 1.80  | 1.53  | 1.92  | 2.01  | 1.95  | 1.97  |
| After 6th 14 days.....          | 2.04  | 1.80  | 1.98  | 1.99  | 1.92  | 2.17  |
| After 7th 14 days.....          | 1.64  | 1.29  | 1.49  | 1.71  | 1.63  | 1.57  |
| Average.....                    | 1.47  | 1.36  | 1.63  | 1.65  | 1.57  | 1.60  |

## ANTIBIOTICS FROM OTHER STREPTOMYCES

### Viridogrisein Plus Griseoviridin

E.L.R. Stokstad and E.C. De Renzo (U.S. Patent 2,929,711; March 22, 1960; assigned to American Cyanamid Company) discovered that the antibiotics called viridogrisein and griseoviridin, which are produced by certain unidentified species of *Streptomyces* and by the certain strains of *Streptomyces griseus*, may be effectively used in stimulating the growth of animals when incorporated in their feed in small amounts.

Of these two antibiotics, viridogrisein and griseoviridin, the former appears to be more effective in stimulating the growth of animals when the purified materials are incorporated in the animals' feed. Surprisingly, when both antibiotics are included, there appears to be a synergistic effect whereby greater growth is obtained with the same amount of feed in the same period of time than would be the case when using either of the others alone. This remarkable synergistic action is demonstrated by the following.

## Antibiotics as Anabolic Stimulators

Example: A basal poultry diet was prepared. One-day-old chicks were used in this series of experiments. Forty-eight baby chicks were used as controls and two groups of twelve chicks were used for each of the diets. The birds were given feed and water ad libitum.

| <u>Supplement</u>           | <u>Weight in grams<br/>at 19 days</u> |
|-----------------------------|---------------------------------------|
| None                        | 143                                   |
| Procaine penicillin, 10 mg. | 145                                   |
| Viridogrisein—              |                                       |
| 3 mg.                       | 150                                   |
| 10 mg.                      | 152                                   |
| Griseoviridin—              |                                       |
| 3 mg.                       | 142                                   |
| 10 mg.                      | 149                                   |
| Viridogrisein, 1.5 mg. }    |                                       |
| Griseoviridin, 1.5 mg. }    | 161                                   |
| Viridogrisein, 5 mg. }      |                                       |
| Griseoviridin, 5 mg. }      | 179                                   |

The foregoing experiments were made with essentially pure viridogrisein and griseoviridin. Another series was run in which the antibiotics were contained in a material which consisted of a dried aqueous fermentation broth which had been fermented with a species of *Streptomyces* sp. T-2184 which is known to produce both viridogrisein and griseoviridin, the former being present in predominating proportions. This fermentation liquor had been filtered to remove mycelia and was then frozen and dried to obtain a lyophilized product. Again there were favorable growth promoting results when this was used.

### Staphylomycin

Staphylomycin, as well as concentrates and mycelia from the broth of *Streptomyces virginiae* 899 (ATCC No. 13161) are found to be growth promoting additives (5 to 50 g./ton swine feed, and 2 to 50 g./ton poultry feed) by P. Van Dijck and H. Eyssen (U.S. Patent 3,017,272; January 16, 1962; assigned to Recherche et Industrie Therapeutiques, Belgium).

Example 1: A basal feed mixture of a standard 16% protein, corn, soybean meal ration fortified with vitamins and minerals combined with additives as shown below was fed to two replicates of 5 Hampshire swine at each level for 69 days. All weights are expressed in pounds.

(a) Basal ration with no additive: total gain = 729 lbs.; total average daily gain = 1.16 lbs.; total feed consumed = 2,325 lbs.; feed efficiency for two replicates = 3.18 pounds per pound gain.

(b) Basal ration with 40 g. of Aureomycin per ton: total gain = 873 lbs.; total average daily gain = 1.26 lbs.; total feed consumed = 2,724 lbs.; feed efficiency for two replicates = 3.12 pounds per pound gain.



## Antibiotics as Anabolic Stimulators

(c) Basal ration with 20 g. of Staphylomycin per ton: total gain = 911 lbs.; total average daily gain = 1.316 lbs.; total feed consumed = 2,658 lbs.; feed efficiency for two replicates = 2.92 pounds per pound gain.

(d) Basal ration with 40 g. of Staphylomycin per ton: total gain = 1,019 lbs.; total average daily gain = 1.476 lbs.; total feed consumed = 2,927 lbs.; feed efficiency for two replicates = 2.87 pounds per pound gain.

Example 2: Broiler chicks were fed a basal diet consisting of proteins, 20.25% and starch value, 68.92%. Each group contained 50 animals.

| 3 days wt.                        | 17 days wt. | 31 days wt. | Percent increase over controls | Feed Efficiency (gms. per gm. gain) |
|-----------------------------------|-------------|-------------|--------------------------------|-------------------------------------|
| BASAL FORMULA WITH NO ADDITIVE    |             |             |                                |                                     |
| 45.8                              | 152.6       | 332         | -----                          | 2.704                               |
| STAPHYLOMYCIN (10 GMS. PER TON)   |             |             |                                |                                     |
| 46.0                              | 163.4       | 376         | 15.3                           | 2.523                               |
| OXYTETRACYCLINE (10 GMS. PER TON) |             |             |                                |                                     |
| 46.3                              | 159.0       | 346         | 4.8                            | 2.629                               |
| PENICILLIN (4 GMS. PER TON)       |             |             |                                |                                     |
| 46.3                              | 158.8       | 348         | 5.5                            | 2.761                               |

### Spiramycins

C.R.M. Schuppon (U.S. Patent 3,039,874; June 19, 1962; assigned to Societe des Usines Chimiques Rhone-Poulenc, France) found that the antibiotic substances produced by the culture of *Streptomyces ambofaciens*, and designated Spiramycins I, II, and III, as well as mixtures of these Spiramycins possess anabolic properties.

Example 1: Two groups of 11 heifers each aged about one year and weighing about 200 kg. were raised on pasture, but the heifers of one group received each day, in addition, Spiramycin (100 mg.) orally. The results were as follows:

|                              | Control group, kilograms | Treated group, kilograms |
|------------------------------|--------------------------|--------------------------|
| Initial weight of group----- | 2,190                    | 2,084                    |
| After 12 weeks-----          | 2,284                    | 2,277                    |
| Gain in 12 weeks:            |                          |                          |
| Per group-----               | 94                       | 193                      |
| Per individual-----          | 8.540                    | 17.540                   |

Example 2: Trials are carried out with Hampshire and Landrace pigs divided into groups of 10 to 14 with 5 groups per dosage rate; in all a total of 59 pigs per dosage rate. In each group the piglets were 5.5 to 5.750 kg. in weight and 28 to 35 days old. They were chosen in such a way that in each series of treatments the groups receiving the different treatments

## Antibiotics as Anabolic Stimulators

were completely comparable, having the same number of piglets, the same average weight and the same average age. The trial lasted 28 days in the case of the first series of treatments and for 35 days in the case of the four other series.

The basic ration was composed of 56.5% maize and 21% soya bean, with 2.5% "fish solubles" and 15% skimmed-milk powder as source of animal protein. In addition there were present stabilized fat (2.5%), vitamins, mineral salts and saccharin (0.05%). The ration contained 18% crude protein. The results were as follows:

|   | Control | Spiramycin       |                |                |
|---|---------|------------------|----------------|----------------|
|   |         | 12.5<br>g./tonne | 25<br>g./tonne | 50<br>g./tonne |
| Average weight per subject (kg.):   |         |                  |                |                |
| at commencement.....  | 5.53    | 5.75             | 5.66           | 5.75           |
| after 15 days.....  | 7.07    | 7.83             | 8.06           | 8.20           |
| after 28 to 35 days.....  | 10.33   | 11.86            | 13.63          | 14.13          |
| Total gain in weight per subject (kg.):   |         |                  |                |                |
| During the first 15 days..  | 1.54    | 2.08             | 2.40           | 2.45           |
| Total.....  | 4.8     | 6.11             | 7.97           | 8.38           |
| Increase in growth rate (percent compared with control):                                    |         |                  |                |                |
| during first 15 days, percent.....  |         | +35              | +55            | +59            |
| Total, percent.....   |         | +27              | +66            | +74            |
| Feed conversion (or average weight of food (kg.) per kilogramme gain in weight), total..... | 2.89    | 2.40             | 1.95           | 1.83           |
| Saving in food compared with control, percent.....  |         | 17               | 32             | 36             |
| Death rate, percent.....  | 8.5     | 5.1              | 1.7            | 3.4            |

### Neomycin Resin Adsorbate

G. St. Clair (U.S. Patent 3,250,623; May 10, 1966; assigned to S.B. Penick and Company) found that neomycin in the form of the carboxylic acid resin-adsorbate gives improved weight gain and feed utilization in livestock and poultry. The neomycin resin-adsorbate is described in U.S. Patent 3,085,936. It is formed by the base-exchange reaction between a neomycin solution and a carboxylic acid resin. The neomycin solution can be obtained from solutions of salts or the base of neomycin or as a result of the growth of a neomycin-producing strain of *Streptomyces fradiae* in an aqueous nutrient medium.

**Example 1:** Chickens - Two flocks of approximately 6,000 chickens each, at four and a half weeks of age were vaccinated for Newcastle disease and laryngotracheitis. This necessitated penning and catching the birds, putting the entire flock under a severe stress. Reaction to vaccination, together with this stress of catching, generally results in a respiratory condition which leads to chronic respiratory disease. Each group was put on a booster feed for three days. The first group was fed a chlortetracycline feed and the second group was given the same containing 100 grams of the neomycin adsorbate per ton of feed. The results are shown below.

|  | Control | Treated |
|--|---------|---------|
| Number of birds.....   | 6,300   | 5,700   |
| Final weight (pounds).....   | 3.78    | 3.74    |
| Feed Conversion efficiency, i.e., Pounds feed consumed : Pounds weight gained..... | 2.43    | 2.31    |
| Mortality (number).....  | 36      | 18      |



## Antibiotics as Anabolic Stimulators

**Example 2:** Pigs - Seventy-five pigs from 11 litters were weighed and divided by weight and litter into five lots of 15 pigs each. All the pigs were between two and three weeks of age and the lots were balanced according to weight with an average weight of about 12 lbs. One control lot was fed a basal ration containing no antibiotic. Two lots were fed the neomycin resin-adsorbate at a level of 200 grams of neomycin activity per ton and two lots were fed the resin adsorbate at a level of 100 grams of neomycin activity per ton. When fed at the 200 gram neomycin activity level for twenty-five days, the average gains in the two lots were 60% greater than the negative controls. The average feed required per pound of gain in the lots at this level was 11.2% less than in the negative controls. At the 100 gram per ton level, gains in the two lots of 15 pigs was increased 53% over the negative controls and the feed required per pound of gain in the medicated lots was 11.8% less than the control lot. No deaths occurred in the pigs fed at the higher level. One death occurred at the 100 gram level. Two pigs died in the control lot.

### Moenomycin

F. Bauer, G. Huber and K.H. Wallhäuser (U.S. Patent 3,279,923; October 18, 1966; assigned to Farbwerke Hoechst AG, Germany) found that moenomycin containing feedstuffs (0.5 to 20 g./ton) promote growth and improve feed efficiency.

**Example:** A basic chick feed mixture containing vitamins and minerals was fed to 80 Nicholas chicks with added antibiotics for a period of 6 weeks. Results are tabulated below.

| Additive                | Quantity of antibiotic in mg. per kg. of feed | After 2 weeks             |                                 |                         |   | After 4 weeks             |                                 |                         |   | After 6 weeks             |                                 |                         |   |
|-------------------------|---|---------------------------|---------------------------------|-------------------------|---|---------------------------|---------------------------------|-------------------------|---|---------------------------|---------------------------------|-------------------------|---|
|                         |   | Average weight gain in g. | Improved weight gain in percent | Average feed efficiency | Improvement of feed efficiency in percent | Average weight gain in g. | Improved weight gain in percent | Average feed efficiency | Improvement of feed efficiency in percent | Average weight gain in g. | Improved weight gain in percent | Average feed efficiency | Improvement of feed efficiency in percent |
| None (Control).....     | -----   | 139.5                     | -----                           | 1.85                    | -----                                     | 515.3                     | -----                           | 2.14                    | -----                                     | 963.9                     | -----                           | 2.47                    | -----                                     |
| Chlorotetracycline..... | 10  | 139.5                     | 0                               | 1.81                    | +2.2                                      | 509.8                     | -1.1                            | 2.11                    | +1.4                                      | 967.0                     | +0.3                            | 2.40                    | +2.9                                      |
| Penicillin.....         | 10  | 137.5                     | -1.5                            | 1.77                    | +4.5                                      | 508.0                     | -1.4                            | 2.09                    | +2.4                                      | 987.9                     | +2.5                            | 2.39                    | +3.3                                      |
| Moenomycin.....         | 10  | 155.3                     | +11.3                           | 1.77                    | +4.5                                      | 551.6                     | +7.0                            | 2.08                    | +2.9                                      | 1,010.1                   | +4.8                            | 2.36                    | +4.7                                      |

### Phytoactin and Phytostreptin

J. Ziffer made the surprising discovery that the primarily antifungal substances polyamido-hygrostreptin (Phytoactin) and polyaminohygrostreptin (Phytostreptin), are both nutritionally and therapeutically beneficial when administered to animals. (U.S. Patent 3,155,520; November 3, 1964; assigned to Pabst Brewing Company). Both are produced by the cultivation of a strain of the species Streptomyces hygroscopicus.

A culture of a microorganism strain which was isolated from domestic United States soil and produces Phytoactin has been deposited in the culture collection of the United States Department of Agriculture, Agricultural Research, Northern Utilization Research and Development Division, Peoria, Illinois, and the culture has been assigned the number NRRL 2752 in the culture collection. The strain which produces Phytostreptin has been assigned the number NRRL 2751. Both are distinct from other antifungal, antibacterial antibiotics previously reported.



Production of Antifungals by Fermentation: The antifungals are produced by fermenting a nutrient medium with a Phytoactin or Phytostreptin producing microorganism such as Streptomyces hygroscopicus NRRL 2752 or 2751, respectively, under submerged, aerobic and agitated conditions until substantial antifungal activity is produced.

Nutrient media include a suitable source of assimilable carbon, such as glucose, a source of assimilable nitrogen such as soya flour, corn steep liquor, yeast and the like, and mineral salts, which may be present with the other ingredients, such as corn steep liquor. Inoculum of the organism is prepared by growing it on agar slant media such as oatmeal or peptone-yeast extract. These agar slant cultures can then be used to prepare larger amounts of inoculum by seeding shake flasks containing such media as soya flour and corn steep liquor. These flasks are shaken under conditions suitable for the growth of the organism. Aseptic conditions must be maintained during the preparation of the inoculum and during the subsequent fermentation.

In the fermentation, the desired media is prepared and the pH of the medium adjusted to about 6.7 to 7.2. Calcium carbonate is included in the preferred medium. The medium so prepared is sterilized by heating at an elevated temperature under pressure, i.e., at about 120°C. The medium is then cooled to a temperature of 27° to 34°C. The sterile medium is then inoculated under aseptic conditions with the inoculum prepared as described above.

The fermentation then proceeds at a temperature in the foregoing ranges with agitation and aeration using sterile air, supplied at the rate of about 0.25 to 1.5 volumes of free air per volume of medium per minute. The fermentation is continued for a period time sufficient to achieve optimal and preferably maximal production of Phytostreptin or Phytoactin as the case may be. A fermentation period of 48 to 96 hours is ordinarily sufficient.

The antifungal may be recovered by a number of methods or, alternatively, the whole culture or whole broth may be used as such or may be concentrated or dried by suitable means. It is ordinarily preferred to recover the antifungal by precipitation or by solvent extraction of the whole culture or whole broth. In the precipitation recovery method, the whole culture is usually filtered or centrifuged at a preferred pH range of 7 to 8, and the filtrate is acidified to a preferred pH range of 3 to 5 to precipitate the antifungal. The preferred acid for this precipitation step is hydrochloric acid. Since the culture mycelium contains appreciable quantities of the antifungal, the whole culture (without filtration) may, alternatively, be adjusted to pH 3 to 5 for the precipitation step.

The activity may be recovered from the precipitate or sediment by extraction with a suitable organic liquid in which it is soluble, such as methanol, ethanol, isopropanol, butanol, acetone or methylisobutyl ketone. The solvent solution may then be evaporated in vacuo, and the resulting residue further extracted with organic solvents. In the preferred method of recovery, the latter residue after evaporation is extracted exhaustively with methylisobutyl ketone, and the solvent solution is concentrated in vacuo. The antifungal may then be precipitated by the addition of 5 volumes of diethyl ether. The antifungal remaining in the mother liquor may be recovered by concentrating to small volume in vacuo and adding 5 volumes of petroleum ether (30° to 60°C.) to precipitate the activity. A solvent extract of the whole culture, whole broth or active precipitated sediment may also be used as such after concentration in vacuo.



## Antibiotics as Anabolic Stimulators

Data at the end of 8 weeks is given below.

Example 1: Day-old Cornish-Arbor Acre Cross hatchery run chicks were divided into four groups of approximately equal average weight, 30 chickens per group.

| Supplement added         | Equivalent<br>Phytoactin<br>activity, gms.<br>per ton feed | Average<br>chick<br>weights<br>(gms.) | Average<br>chick<br>weight<br>gains<br>(gms.) | Average<br>feed<br>efficiency |
|--------------------------|--|---------------------------------------|---|-------------------------------|
| Basal ration only-----   | -----  | 1,293                                 | 1,249   | 2.32                          |
| Purified Phytoactin----- | 2  | 1,356                                 | 1,313   | 2.28                          |
| Purified Phytoactin----- | 10   | 1,369                                 | 1,325   | 2.32                          |
| Purified Phytoactin----- | 25   | 1,353                                 | 1,310   | 2.20                          |

Example 2: Day-old Vantress hatchery run chicks were divided into two groups, 21 chicks per group, with one group receiving only the basal ration, and the remaining group receiving the basal ration supplemented with Phytoactin dried fermentation whole culture (D.F.W.C., 4.54 g. Phytoactin per pound).

| Supplement added        | Equivalent<br>Phytoactin<br>activity, gms.<br>per ton feed | Average<br>chick<br>weights<br>(gms.) | Average<br>chick<br>weight<br>gains<br>(gms.) | Average<br>feed<br>efficiency |
|-------------------------|--|---------------------------------------|---|-------------------------------|
| Basal ration only-----  | -----  | 1,225                                 | 1,181   | 2.38                          |
| Phytoactin, D.F.W.C.--- | 10   | 1,275                                 | 1,230   | 2.43                          |

Example 3: Day-old White Rock hatchery chicks were divided into two groups, 30 chicks per group, with one group receiving only the basal ration, and the remaining group receiving the basal ration supplemented with Phytostreptin dried fermentation whole culture (11.4 g. Phytostreptin per pound).

| Supplement added           | Equivalent<br>Phytostreptin<br>activity,<br>gms. per ton<br>feed | Average<br>chick<br>weight,<br>gms. | Average<br>chick<br>weight<br>gains,<br>gms. | Average<br>feed<br>effi-<br>ciency |
|----------------------------|--|-------------------------------------|--|------------------------------------|
| Basal ration only-----     | -----  | 1,223                               | 1,183  | 2.38                               |
| Phytostreptin, D.F.W.C.--- | 10   | 1,285                               | 1,245  | 2.37                               |

### Oleandomycin

W.C. Sherman, G.A. Donovan, W.M. Reynolds, and H.G. Luther (U.S. Patent 2,925,342; February 16, 1960; assigned to Chas. Pfizer and Company) found that animal feeds containing nutritional levels of the antibiotic, oleandomycin, elicit a substantial growth increase and feed efficiency response. The oleandomycin can be in the form of free base, pharmacologically acceptable acid addition salts or biologically active derivatives.

Example: The growth experiments with oleandomycin were conducted on Nichols white-cross chicks kept in electrically heated brooders on raised wire floors. The day old chicks were divided into lots of five males and five females per compartment, replicated twice

## Antibiotics as Anabolic Stimulators

per treatment, and fed a basal diet containing 56% ground yellow corn. The antibiotic test materials were added to the diet in premix form at the expense of yellow corn meal.

| Treatment           | Antibiotic,<br>grams/ton | Av. Weight<br>(grams) |         | Feed Efficiency<br>(lbs. Feed/lbs. Gain)<br>4 weeks | Mortality,<br>Percent |
|---------------------|--------------------------|-----------------------|---------|---|-----------------------|
|                     |                          | 2 weeks               | 4 weeks |   |                       |
| None                | —                        | 170                   | 425     | 1.64  | 2.5                   |
| Procaine Penicillin | 5                        | 175                   | 431     | 1.61  | 2.5                   |
| Procaine Penicillin | 25                       | 178                   | 440     | 1.61  | 0                     |
| Oleandomycin base   | 5                        | 185                   | 455     | 1.60  | 0                     |
| Oleandomycin base   | 25                       | 188                   | 461     | 1.60  | 2.5                   |

As illustrated above, it was found that the inclusion of procaine penicillin at the rate of five or 25 grams per ton produced only slight, nonsignificant improvements in growth and feed efficiency. However, equal levels of oleandomycin produced growth responses of approximately three times the magnitude obtained with procaine penicillin. The superiority of the growth with oleandomycin over that with procaine penicillin was statistically significant.

### Oleandomycin Resin Adsorbate

E. G. Martin and W. J. Haas (U. S. Patent 2,970,053; January 31, 1961; assigned to Chas. Pfizer and Company) found that oleandomycin resin adsorbates, wherein the oleandomycin is adsorbed on strong acid cation-exchange resins such as cation-exchange resins containing sulfonic acid groups, exhibit unusual stability. The resins include Dowex 50-X1 and X2, Amberlite IR-120 and XE-176.

Example 1 - Preparation: Amberlite XE-176 ion-exchange resin in the sodium cycle was washed with water, dried at 100°C. and ground to a fine powder. Material passing through a 200 mesh screen was separated for use. 40 gallons of water extract containing oleandomycin phosphate equivalent to a potency of 10,000  $\gamma$ /ml. of oleandomycin base was prepared. To the aqueous solution was added 1,745 grams of the powdered resin. The mixture was stirred for 2 hours and the resin filtered and washed. The resin cake was repulped in 10 gallons of water, refiltered, washed and dried at 35° to 50°C. in a vacuum oven. The dried resin adsorbate was found to have an activity equivalent to 400 mg. of oleandomycin base per gram of dry resin adsorbate.

Stability studies on animal feeds containing this resin adsorbate were conducted over a period of several weeks and compared with stability data of several other forms of oleandomycin obtained under identical conditions. For convenience, the oleandomycin salts were premixed with Supercel as diluent then added to the animal feed in amounts ranging from about 400 to about 650 mg. of oleandomycin per gram of feed. These relatively high concentrations were utilized to facilitate assay of the samples. Samples were stored at various temperatures and assayed periodically for their biopotency. The resin adsorbate even after 6 weeks at 50°C. retained greater than 55% of its potency whereas compositions of the other salts lost more than 60% of their potency within 3 weeks.



## Antibiotics as Anabolic Stimulators

| <u>Oleandomycin Salt</u> | <u>Initial<br/>Assay</u> | <u>3 Weeks<br/>50°</u> | <u>6 Weeks</u> |            |
|--------------------------|--------------------------|------------------------|----------------|------------|
|                          |                          |                        | <u>25°</u>     | <u>50°</u> |
| Lauryl sulfate           | 0.456                    | 0.182                  | 0.369          |            |
| Methylene disalicylate   | 0.387                    | 0.054                  | 0.393          |            |
| Chloroform               | 0.387                    | 0.033                  | 0.193          |            |
| Trichloroethane          | 0.537                    | 0.037                  | 0.272          |            |
| Tannate                  | 0.483                    | 0.224                  | 0.394          |            |
| Resin Adsorbate          | 0.655                    | 0.592                  | 0.522          | 0.362      |

Example 2 - Use: The inclusion of oleandomycin resin adsorbate in the nutritionally balanced basal diet of day-old chicks was found to produce substantial improvements in growth (4 week weights), and feed efficiency over that of the negative control group and the procaine penicillin supplemented group.

### Spectinomycin

M. E. Bergy and C. De Boer (U.S. Patent 3,245,797; April 12, 1966; assigned to The Upjohn Company) found an increased rate of growth and/or an enhanced utilization of feedstuff can be produced by feeding spectinomycin in combination with the feed or drinking water to healthy animals.

The concentration of spectinomycin in the feed composition is determined with regard to the species of animal, age, weight, and average amount of feed consumed daily. The following table illustrates the range of spectinomycin in grams per ton of feed for representative animals.

| Animal                         | Range<br>(gram/ton) | Preferred<br>amount<br>(gram/ton) |
|--------------------------------|---------------------|-----------------------------------|
| Swine (birth to 8 weeks) ..... | 15-200              | 100                               |
| Swine (40-100 lb.) .....       | 10-200              | 50                                |
| Chicken (0-12 weeks) .....     | 2-100               | 10                                |
| Turkeys (0-24 weeks) .....     | 2-100               | 10                                |
| Beef cattle (fattening) .....  | 2-100               | 8                                 |
| Calves (0-12 weeks) .....      | 10-200              | 50                                |
| Lambs .....                    | 5-100               | 25                                |

Example 1: A fattening feed for 800 pound yearling cattle consists of:

|                                    |       |         |
|------------------------------------|-------|---------|
| Ground ear corn                    | 89.75 | Percent |
| Soybean oil meal, 44%              | 9.0   | Percent |
| Ground limestone                   | 0.7   | Percent |
| Salt                               | 0.5   | Percent |
| Trace mineral mixture <sup>1</sup> | 0.05  | Percent |

<sup>1</sup> Contains the following percent of minerals: Mn, 12; Co, 0.08; Fe, 5.0; Cu, 0.4; I, 0.24; Zn, 0.7

To 999 parts of the preceding feed is added 1 part of a premix composition prepared by mixing 4 g. of spectinomycin free base with sufficient wheat flour to make 1 pound. The feeding composition so prepared supplied 4 mg. of spectinomycin per pound, or about 8.8

## Antibiotics as Anabolic Stimulators

parts per million. Cattle are to receive the foregoing feed ad libitum together with 5 lbs. of hay per head per day and when so fed have increased rate of weight gain and improved utilization of feed.

Example 2: A premix for addition to drinking water is prepared from the following types and amounts of ingredients:

|                             |           |
|-----------------------------|-----------|
| Spectinomycin hydrochloride | 4 grams   |
| Sucrose                     | 450 grams |

The spectinomycin hydrochloride, and sucrose are mixed together. The premix is added to drinking water in the following amounts: chickens and turkeys 1/2 lb./250 gallons; and beef cattle 1 lb./250 gallons. Spectinomycin can be used in combination with other antibiotics (1 to 4 parts of spectinomycin to 1 part of neomycin, penicillin, novobiocin, and erythromycin).

### Combination of Spectinomycin and Lincomycin

M.E. Bergy and R.R. Herr (U.S. Patent 3,261,687; July 19, 1966; assigned to The Upjohn Company) found that the results achieved by use of the antibiotic combination in association with an animal feed are greater than can be achieved by use of any single member. The antibiotics are added to the animals' nutritionally adequate feedstuff in a total amount of from 5 to 100 mg. per pound of feed. The antibiotics are present in a ratio of from 1 part to 10 parts of spectinomycin to 1 part of lincomycin. Preferred is 3 parts of spectinomycin to 1 part of lincomycin.

The following table illustrates the range of combined lincomycin and spectinomycin in grams per ton of feed for representative animals.

| Animal                        | Range<br>(gram/ton) | Preferred<br>amount<br>(gram/ton) |
|-------------------------------|---------------------|-----------------------------------|
| Swine (birth to 8 weeks)..... | 15 to 200           | 75                                |
| Swine (40-100 lb.).....       | 10 to 200           | 50                                |
| Chicken (0-12 weeks).....     | 4 to 100            | 10                                |
| Turkeys (0-24 weeks).....     | 4 to 100            | 10                                |
| Beef cattle (fattening).....  | 4 to 100            | 8                                 |
| Calves (0-12 weeks).....      | 10 to 200           | 50                                |
| Lambs.....                    | 5 to 100            | 25                                |

The concentration of the antibiotics in water is about 1/2 the concentration (weight to weight basis) of the antibiotic concentration in feed. For increased production in meat-producing animals in an infectious diseased state the preferred concentration of antibiotics in the feed can be increased for the various species to the following levels: swine, birth to 8 weeks, 400 g./ton; swine, 40 to 100 lbs., 400 g./ton; chicken, up to 12 weeks, 800 g./ton; turkeys, up to 24 weeks, 800 g./ton; beef cattle, fattening, 200 g./ton; calves, up to 12 weeks, 400 g./ton; and lambs, 200 g./ton. The animal feeds containing the increased amounts of antibiotics are fed for a shorter time during the period when therapeutic treatment is required.

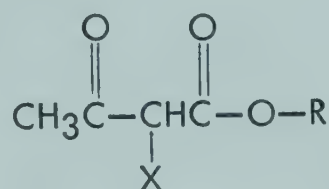


Example - Premix Composition:

|                          |          |
|--------------------------|----------|
| Lincomycin hydrochloride | 0.1 gram |
| Spectinomycin sulfate    | 0.3 gram |
| Calcium carbonate, q.s.  | 1.0 lb.  |

ANTIMICROBIALSAlkyl-2-Haloacetoacetates

The process developed by M. Legator (U.S. Patent 3,148,988; September 15, 1964; assigned to Shell Oil Company) provides for the oral administration of antimicrobial compounds to living animals in order to stimulate, accelerate or promote the growth of an animal. These growth promotants are alkyl-2-haloacetoacetates which may be represented by the following structural formula:



wherein R represents branched or straight-chain alkyl group, a substituted (preferably substituted with a halogen atom or NO<sub>2</sub> group) alkyl group, a cycloalkyl group or a substituted cycloalkyl group. When R is a straight-chain or cycloalkyl group, it preferably contains 1 to 20 carbon atoms. X represents a halogen atom, preferably chlorine. Of special interest are the alkyl 2-chloroacetoacetates wherein R represents a straight-chain alkyl of 1 to 5 carbon atoms, e.g., methyl 2-chloroacetoacetate.

Example 1: Four-week, six-week, eight-week growth promotant results in lambs: The following table illustrates growth stimulation in lambs with methyl 2-chloroacetoacetate. This growth stimulant was orally administered to the lambs by mixing the promotant with the feed. Ten lambs were used per treatment with the exception of the check and the aureomycin standard where eight lambs were used. The lambs at the start of the test weighed about 80 pounds each and were from about 4 to about 5 months old.

| Compound                         | Quantity Per Lamb Per Day in Feed, mg. | Lb. Gain Per Animal Per Day at— |        |        | Pounds Feed Consumed Per Pound Gain (Feed Conversion Ratio) |
|----------------------------------|--|---------------------------------|--------|--------|---|
|                                  |  | 4 Wks.                          | 6 Wks. | 8 Wks. |   |
| Methyl 2-chloroacetoacetate..... | 50                                     | .40                             | .37    | .40    | 10.6  |
| Aureomycin.....                  | 25                                     | .43                             | .39    | .37    | 11.4  |
| Check.....                       | -----                                  | .21                             | .26    | .29    | 12.0  |

It can be seen from the table that the weight gain per animal was fairly constant for the eight-week period. This is in contrast to aureomycin where the weight gain decreased after the first four weeks.

Methyl 2-chloroacetoacetate, although somewhat inferior to aureomycin at the four-week weighing, was slightly better in weight gain and feed conversion when the test was terminated. There were no appreciable differences in the grade of the carcasses from any of the treatments, and all appeared slightly better than the checks. From the previous table it can be seen that the test material was equal or superior to the aureomycin standard in both weight gain and feed conversion ratio.

Example 2: Sixty-day growth promotant results in lambs. Methyl 2-chloroacetoacetate was blended into a standard feed and fed throughout a sixty-day test period at a dosage of 100 mg. per lamb per day. Aureomycin was the standard at 25 mg. per lamb per day. Ten feeder lambs weighing about 80 pounds each were used for each treatment. Methyl 2-chloroacetoacetate at 100 mg. per lamb per day gave a 25% improvement over the control.

### Acroleins and Beta-Methoxypropionaldehyde

This later process of M. Legator (U.S. Patent 3,240,605; March 15, 1966; assigned to Shell Oil Company) is again based on the oral administration of antimicrobial compounds to living animals in order to stimulate, accelerate or promote the growth of the animal.

The described growth promoters are: (1) Alpha,beta-olefinically unsaturated aliphatic aldehydes, especially the alpha-methylidene alkenal, acrolein. (2) Alpha,beta-alkenal dialiphatic acetals, especially acrolein dialiphatic acetals as represented by acrolein dimethyl acetal. (3) Beta-aliphaticoxyaldehydes, especially beta-alkoxypropionaldehydes, as represented by beta-methoxypropionaldehyde.

Example 1: Four-week, six-week, eight-week growth promotant results in lambs: These growth stimulants were in each case orally administered to the lambs by mixing the promotant with the feed or, in the case of acrolein, adding it to the drinking water daily. Ten lambs were used per treatment with the exception of the check and the aureomycin standard where eight lambs were used. The lambs at the start of the test weighed about 80 pounds each and were from about 4 to 5 months old.

| Compound                              | Quantity<br>Per Lamb<br>Per Day in<br>Feed or<br>Water, mg. | Lb. Gain Per<br>Animal Per Day<br>at— |           |           | Pounds Feed<br>Consumed<br>Per Pound<br>Gain (Feed<br>Conversion<br>Ratio) |
|---------------------------------------|---|---------------------------------------|-----------|-----------|--|
|                                       |   | 4<br>Wks.                             | 6<br>Wks. | 8<br>Wks. |  |
| Acrolein.....                         | 100   | .43                                   | .36       | .36       | 10.5   |
| Acrolein dimethyl<br>acetal.....      | 50  | .35                                   | .35       | .36       | 11.4   |
| Beta-methoxypropion-<br>aldehyde..... | 50  | .30                                   | .41       | .42       | 10.2   |
| Aureomycin.....                       | 25  | .43                                   | .39       | .37       | 11.4   |
| Check.....                            |   | .21                                   | .26       | .29       | 12.0   |

It can be seen from the table, that the weight gain per animal was fairly constant for the eight-week period. This is in contrast to aureomycin where the weight gain decreased after the first four weeks.

Acrolein and aureomycin were similar in results. The decrease in the rate of weight gain on treatment with antibiotics, such as shown, is attributed to the establishment of a resistant



flora after prolonged use. The evidence currently available seems to indicate that the growth promotants of this process are not beset with this problem. The reason for a similar decline in rate with acrolein after the fourth week is not definitely known, but believed to be attributed to the loss of acrolein from the water trough by vaporization so that the consumption was actually less than indicated. The results with acrolein are complicated by variations in the amount consumed by the animals. Although acrolein was added fresh daily to the water trough, the rate of loss from the water varied with conditions. On a day when the temperature reached 100°F. in the late afternoon, it was initially found, by chemical analysis, that there were 22 ppm in the water trough at 8:30 a.m., but only 12 ppm at 4:00 p.m.

Acrolein dimethyl acetal was the poorest of the materials evaluated in this test but nevertheless about equal to the aureomycin standard. Although beta-methoxypropionaldehyde at the four-week weighing showed very little increase over a normally anticipated gain of 0.30 to 0.35 lb. per day, a sharp increase in weight gain occurred between the fourth and sixth week weighing, placing this compound first in this particular test.

There were no appreciable differences in the grade of the carcasses from any of the treatments, and all appeared slightly better than the checks. From the previous table, it can be seen that all the test materials were equal or superior to the aureomycin standard in both weight gain and feed conversion ration.

Example 2: Sixty-day growth promotant results in lambs: Beta-methoxypropionaldehyde was blended into a standard feed and fed throughout a 60-day test period at a dosage of 50 and 100 mg./lamb/day. Aureomycin was the standard at 25 mg./lamb/day. Ten feeder lambs weighing about 80 pounds each were used for each treatment.

An effective treatment was obtained with beta-methoxypropionaldehyde at 100 mg./lamb/day. This compound increased daily weight gain about 33% over the untreated controls and was equal to or slightly better than the aureomycin standard.

## MISCELLANEOUS

### Curvularin

H.L. Wehrmeister (U.S. Patent 3,436,223; April 1, 1969; assigned to Commercial Solvents, Inc.) discovered that in meat-producing animals an increased rate of growth and an increased efficiency of food conversion can be achieved by administering curvularin to such animals either in combination with a conventional feed or by administration in a suspending agent, solid carrier, or other vehicle. Curvularin and its production are described in an article entitled, "Curvularin Formation by Penicillium Stechii" by D. Fennell, K.B. Raper and F.H. Stodola appearing in "Chemistry and Industry" (1959) at page 3146.

The amount of curvularin administered depends upon the species of animal, its age, weight, and average amount of feed consumed daily. Ordinarily the dosage administered falls in the range of 0.1 to 20 milligrams, preferably 0.01 to 0.05 milligrams per kilogram body weight of the animal per day.

## Antibiotics as Anabolic Stimulators

The following table illustrates the daily dosage for representative animals.

| <u>Animal</u> | <u>Daily dosage</u> |
|---------------|---------------------|
| Beef cattle   | 5-15 mg.            |
| Lambs         | 1-5 mg.             |
| Swine         | 1-5 mg.             |
| Poultry       | 0.2-3 mg.           |

Example: A fattening feed for 800 pound yearling cattle is prepared from the following types and amounts of ingredients:

|                                    |               |
|------------------------------------|---------------|
| Ground ear corn                    | 89.75 percent |
| Soybean oil meal, 44%              | 9.0 percent   |
| Ground limestone                   | 0.7 percent   |
| Salt                               | 0.5 percent   |
| Trace mineral mixture <sup>1</sup> | 0.05 percent  |

<sup>1</sup> Contains the following percent of minerals: Mn, 12; Co, 0.08; Fe, 5.0; Cu, 0.4; I, 0.24; Zn, 0.7.

To 999 parts of the preceding feed is added 1 part of a premix composition prepared by mixing 2 g. of curvularin with sufficient wheat flour to make 1 pound. The feeding composition so prepared supplied 2 mg. of curvularin per pound, or about 4.4 parts per million. Cattle are to receive 5 lbs. of the foregoing feed per head per day together with hay and when so fed have an increased rate of weight gain and improved utilization of feed.

### Hydrolyzates of Mycelia of Actinomycetes *Nocardia Rugosa*

R. Faustini, G. Gasparini, A. Tardani, and R. Barchielli (U.S. Patent 3,266,901; August 16, 1966; assigned to Società Farmaceutici Italia, Italy) discovered that hydrolysates of mycelia of actinomycetes *Nocardia rugosa*, free from vitamin B12, are very effective for promoting body-weight increases in animals and for improving the conversion index of foodstuffs. The velocity of body increase in animals brought about by feeds containing hydrolysates of mycelia of *Nocardia rugosa* is surprisingly superior to that brought about by feeds containing hydrolysed mycelia of *Nocardia rugosa*, or by those containing vitamin B12, or by those containing pure nucleinic acids, and is of the same order of magnitude as those feeds containing antibiotics.

Preparation: A boiler of 80 liters capacity provided with shell for steam heating, a stirrer, a thermometer, and a joint for applying vacuum, was filled with 20 liters of water, 2.6 kg. of solution of 36° Baume sodium hydroxide and 16 kg. of *Nocardia rugosa* exhausted mycelium (composed of 20.89% ash, 14.88% organic substance, and 64.23% water).

The temperature of the mass was raised to 29° to 32°C., and it was kept under stirring for 2 hours. Commercial hydrochloric acid was added ( $D_{20}=1.18$ ) till the pH of the mixture was brought to a value ranging from 5.5 to 6. The liquid was filtered with a filter-press and the filter was washed with water until 65 liters of filtrate were collected. The liquid was again placed into the boiler and concentrated in vacuo, at an internal maximum temperature of



60°C. to 8 liters of residual volume. 8 liters of methanol were added to the residue and it was allowed to stand for at least 2 hours. The boiler was then emptied and the mass not dissolved in methanol was filtered off, dried at 60°C. in vacuo, and finally milled. 0.85 to 0.90 kg. of product free from vitamin B12 was obtained. The same results were obtained when potassium hydroxide was used instead of sodium hydroxide.

## ANTIOXIDANTS IN FEEDS

It is well known that the carotene in dehydrated alfalfa and other forage crops is subject to oxidation and that loss of vitamins, particularly vitamin A, occurs under normal storage conditions. Furthermore, animal feeds which include the dehydrated forage crops and other vitamin containing components may undergo further decomposition, such that animals fed thereon are subject to malnutrition and more serious disabilities attributable to deficiencies in essential vitamins.

It has also been found that many animals have the ability to store vitamins, and the longevity of retention of these vitamins for future use will be extended if antioxidants are included in the animal diets. The animal industry now regularly feeds antioxidants to animals as a feed component. The commercial manufacturers of prepared feeds also include antioxidants if the formulation includes vitamins and vitamin containing components, such as dehydrated alfalfa and other forage crops. Such feeds have the ability to retain their full nutrient qualities through the storage periods incident to normal marketing conditions.

It has been found that certain classes of compounds activate the antioxidant properties of even the best antioxidants. These processes are also concerned with such improved antioxidants.

### "SANTOQUIN" ABSORBED ON MINERAL-VEGETABLE POWDER

The antioxidant 6-ethoxy-1,2-dihydro-2,2,4-trimethylquinoline, supplied under the names "Santoquin" and "Ethoxyquin", has found favor as a protective agent for the carotene content of forage crops, and for the vitamin content of animal feeds. Santoquin is a viscous, orange-brown liquid, of specific gravity 1.030 at 25°C., and boiling point 125°C. at 1 to 2 mm. pressure. It is insoluble in water but soluble in animal and vegetable oils and in a variety of organic solvents. The high viscosity of Santoquin, together with the low concentrations at which this compound is used in animal feeds, make it extremely difficult to obtain a homogeneous, well-distributed mixture of liquid Santoquin with the solid constituents of poultry and livestock feed.

G.E. Ward and S.N. Dereniuk (U.S. Patent 3,155,521; November 3, 1964; assigned to Dawe's Laboratories, Inc.) have found that dry Santoquin compositions containing the desired 10% to 70% of Santoquin and having desirable physical characteristics, such as being stable, nonbleeding, nongreasy, flowable, and easily distributed in feeds, can be prepared



by combining certain vegetable materials and certain mineral powders with Santoquin.

Example: In a laboratory model Patterson-Kelley Twin Shell blender was placed 1.2 pounds of finely ground oat hulls (Cell-Flo). One-half pound of Santoquin (6-ethoxy-1,2-dihydro-2,2,4-trimethylquinoline) was added, and the blender was revolved for 10 minutes, with the intensifier bar operating to assist in obtaining a well-mixed product. The blender was stopped, 0.3 pound of synthetic hydrated calcium silicate in the form of Micro-Cel E was added, and the blender was again operated for 10 minutes. The mixed product was withdrawn from the blender and was found to be a nondusty, free-flowing material very suitable for use as an ingredient in poultry and livestock feed. This product had the composition:

|   | Percent |
|---|---------|
| Finely-ground oat hulls                       | 60      |
| 6-Ethoxy-1,2-dihydro-2,2,4-trimethylquinoline | 25      |
| Micro-Cel E                                   | 15      |

### ACTIVATED DIHYDROQUINOLINES

In the following processes, methods of improving the vitamin protective properties of any and all antioxidants, especially 2,2,4-trimethyl-1,2-dihydroquinolines are disclosed. The antioxidant properties of compounds tested are determined by a standardized procedure using carotene emulsions and microanalytical techniques. In all tests, the 6-ethoxy-2,2,4-trimethyl-1,2-dihydroquinoline is used as a standard for control and its antioxidant activity under identical conditions is assigned a rating of 100; the compound being tested is expressed as a percentage of the antioxidant rating of the standard.

#### Dihydroquinolines Activated with 8-Hydroxyquinoline

G.J. Marco (U.S. Patent 3,278,308; October 11, 1966; assigned to Monsanto Company) found that 8-hydroxyquinoline increased the antioxidant properties of dihydroquinolines.

Example: In these tests a 15 microgram sample of 8-hydroxyquinoline was studied alone and compared with a 15 microgram sample of 6-ethoxy-2,2,4-trimethyl-1,2-dihydroquinoline in a standard carotene emulsion. A sample comprised of 15 micrograms of the latter test compound and 5 micrograms of the 8-hydroxyquinoline was also studied under the same conditions to determine its antioxidant properties. The following table sets forth the observed data.

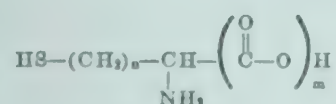
|   | <u>μg.</u> | <u>Antioxidant<br/>Rating<sup>1</sup></u> |
|---|------------|---|
| (A) 6-ethoxy-2,2,4-trimethyl-1,2-dihydroquinoline | 15         | 100                                       |
| (B) 8-hydroxyquinoline                            | 15         | -2.9      3.5                             |
| (A) 15μg. + (B) 5μg.                              | —          | 141.0    156.5                            |

<sup>1</sup> Ratings based on percent of activity of (A) alone, assuming (A) to be 100.

8-Hydroxyquinoline is a chelating agent. Other chelating agents were tested and showed no antioxidant stabilizing effect, hence there is no relationship between chelating power and antioxidant stabilizing effect.

## Dihydroquinolines Activated with Sulfur-Containing Amino Acids

E.G. Jaworski (U.S. Patent 3,279,921; October 18, 1966; assigned to Monsanto Company) found that compounds which have the ability to activate antioxidants have the generic structure:



wherein n is an integer from one to two and wherein m is an integer from zero to one.

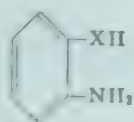
Example: The data set forth below is the percentage of the antioxidant property of the standard antioxidant. The following observations were made:

| Compound   | Alone | 1 to 3 of Standard |
|--|-------|--------------------|
| 6-ethoxy-2,2,4-trimethyl-1,2-dihydroquinoline..... | 100   |                    |
| 1. Cysteine.....                                   | -4.1  | 185.1              |
| 2. 2-aminomercapto butyric acid.....               | -7.6  | 129.3              |
| 3. 2-mercapto ethylamine.....                      | -3.3  | 141.3              |
| 4. 2-hydroxyethylamine.....                        | 1.1   | 111.5              |
| 5. Serine.....                                     | 1.1   | 106                |
| 6. 4-hydroxy-2-amino butyric acid.....             | 0     | 98.5               |
| 7. Cystine.....                                    | 12.2  | 110.8              |
| 8. 2-amino-4-thio-butylolactone.....               | -1.1  | 107.6              |

Of the above Numbers 1, 2 and 3 are included within the scope of the process and are valuable activators for antioxidants. Compounds 4, 5, 6, 7 and 8 have little or no activity and are not included within the scope of the generic definition of useful activators. It is apparent that the replacement of the mercapto group with a hydroxyl radical reduces or destroys the antioxidant activator's property of the adjuvant.

## Dihydroquinolines Activated with Substituted Anilines

E.G. Jaworski (U.S. Patent 3,279,922; October 18, 1966; assigned to Monsanto Company) found that compounds which have antioxidant property includes the compounds of the structure



wherein X is selected from the group of atoms consisting of oxygen and sulfur. The ortho relationship of the substituents is critical; the compounds having the substituents in meta and para positions are not useful as antioxidant activators.



## Antioxidants in Feeds

Example: The data observed is reported as a percent of the activity of the standard both by itself and in combination with the standard antioxidant.

| Compounds   | Alone | 1 : 3 with<br>Standard |
|---|-------|------------------------|
| 6-ethoxy-2, 2, 4-trimethyl-1, 2-dihydroquinoline..... | 100   | -----                  |
| 2-hydroxy aniline.....                                | 17.2  | 140                    |
| 2-mercapto aniline.....                               | 1.1   | 123                    |

### Dihydroquinolines Activated with Nitrogen-Containing Compounds

E.G. Jaworski (U.S. Patent 3,347,677; October 17, 1967; assigned to Monsanto Company) tested the antioxidant activity of many nitrogen-containing compounds. The results are summarized in Table 1.

TABLE 1

|         | Compound   | Antioxidant Activity |   |
|---------|--|----------------------|---|
|         |  | Alone                | 1 part<br>compound<br>plus<br>3 parts<br>standard |
|         | Standard: 6-ethoxy-2,2,4-trimethyl-1, 2-dihydroquinoline.....        | 100                  | -----   |
| 1.....  | Dithiooxamide.....   | -3.0                 | 184.0   |
| 2.....  | N,N'-dimethylthiooxamide.....  | 1.5                  | 138.6   |
| 3.....  | N,N'-dimethyl dithiooxamide.....                                     | 3.0                  | 163.6   |
| 4.....  | Thiooxanilnitrile.....   | 0.9                  | 126.3   |
| 5.....  | O-Zinc salt S-allyl-N-dithiocarboxy-N-phenyl- $\alpha$ -alanine..... | 3.1                  | 132.9   |
| 6.....  | Thioacetanilide.....   | 2.4                  | 165.7   |
| 7.....  | N,N'-dicyclohexyl dithiooxamide.....                                 | 13.6                 | 127.2   |
| 8.....  | Rubeanic acid.....   | 0                    | 187.7   |
| 9.....  | N,N'-bis-2-ethyl mercaptoethyl oxamide.....                          | 3.6                  | 110.7   |
| 10..... | $\alpha$ -Anilino thioacetamide.....                                 | -4.4                 | 126.0   |
| 11..... | Dithiobiuret.....  | -3.3                 | 204.9   |
| 12..... | Thiophthalimide.....   | 0.8                  | 135.8   |
| 13..... | Dithiophthalimide.....   | 2.9                  | 146.9   |
| 14..... | 2-thiopicollnanilide.....  | -1.2                 | 145.7   |
| 15..... | N,N'-bis(2-hydroxyethyl) dithiooxamide.....                          | 2.6                  | 121.5   |
| 16..... | N,N'-bis(carboxymethyl)-dithiooxamide.....                           | 2.7                  | 191.9   |
| 17..... | N,N'-dibenzyl dithiooxamide.....                                     | 4.7                  | 161.0   |
| 18..... | 1,4-piperazine bis(thioacetamide).....                               | 0.5                  | 137.1   |
| 19..... | N(thiocarbamoylmethylene)-p-toluenesulfonamide.....                  | 2.4                  | 161.4   |
| 20..... | 2-dimethylamino-4,6-bis(dimethylthiocarbamyl)-S-triazine.....        | -1.9                 | 240   |
| 21..... | 4-ethoxy-2-thiopicollnanilide.....                                   | -5.2                 | 198.6   |
| 22..... | N,N'-di(3,4-dichlorobenzyl) dithiooxamide.....                       | 3.4                  | 164.0   |
| 23..... | Thiooxanilothioamide.....  | 6.4                  | 139.0   |
| 24..... | N,N'-bis[2(diethylamino)ethyl] dithiooxamide.....                    | 0.9                  | 205.1   |
| 25..... | Dithiobiuret.....  | -4.2                 | 116.5   |
| 26..... | N,N'-didodecyl dithiooxamide.....                                    | 0                    | 128.2   |
| 27..... | N,N'-bis(thiocarbamylmethylene)-p-phenylenediamine.....              | -2.2                 | 110.8   |

The antioxidant activity of various admixtures of rubeanic acid with the 6-ethoxy-2,2,4-trimethyl-1,2-dihydroquinoline standard are given in Table 2.

## Antioxidants in Feeds

**TABLE 2**

| Parts            |                         | Wt. percent<br>RA | Antioxidant<br>Activity Stan-<br>dard=100 |
|------------------|-------------------------|-------------------|---|
| Rubeanic<br>Acid | Standard<br>Antioxidant |                   |   |
| .5               | 15                      | 3                 | 122.4                                     |
| 1.0              | 15                      | 6                 | 123.7                                     |
| 5.0              | 15                      | 25                | 267.1                                     |
| 15               | 15                      | 50                | 293.4                                     |
| 45               | 15                      | 75                | 318.4                                     |
| 225              | 15                      | 94                | 206.6                                     |
| 450              | 15                      | 97                | 159.2                                     |

### Dihydroquinoline Acid Salts

C.C. Tung (U.S. Patent 3,325,288; June 13, 1967; assigned to Monsanto Company) has a process for the preparation of certain salt derivatives of dihydroquinolines which are more effective antioxidants than are the free dihydroquinoline bases.

**Salt Preparation** — A reaction flask was charged with 21.7 grams of 2,2,4-trimethyl-6-ethoxy-1,2-dihydroquinoline and 150 ml. of benzene. The reaction mixture was stirred at 20°C. and 10 grams of 37% aqueous hydrochloric acid (HCl) added. The mixture was refluxed at 81°C. for three hours during which 10 ml. of water were removed from the distillate. The reaction mixture was then cooled to 5°C. and the precipitated solid product was identified as 2,2,4-trimethyl-6-ethoxy-1,2-dihydroquinoline hydrochloride.

**Example 1 — Use of Antioxidants:** The effect of 2,2,4-trimethyl-6-ethoxy-1,2-dihydroquinoline on the stabilization of carotene in alfalfa was studied by storing at 55°C. samples of dehydrated alfalfa treated with dihydroquinoline and its salts. Six replicates of each of the treated alfalfa samples were analyzed before and after the heat treatment. The treated samples of alfalfa included some with 0.015 percent by weight of the free base 2,2,4-trimethyl-6-ethoxy-1,2-dihydroquinoline or the salts.

### % Carotene Left After Two Weeks

|   |      |      |      |      |      |      |      |
|---|------|------|------|------|------|------|------|
| Free base                               | 34.0 | 34.0 | 32.9 | 34.0 | 35.0 | 32.9 | 33.8 |
| HCl salt                                | 38.7 | 36.0 | 38.7 | 36.5 | 37.1 | 35.4 | 37.1 |
| 1:1 H <sub>2</sub> SO <sub>4</sub> salt | 35.4 | 35.4 | 36.0 | 34.3 | 35.4 | 34.3 | 35.1 |
| None                                    | 20.5 | 20.5 | 20.0 | 20.0 | 22.3 | 21.3 | 20.7 |

<sup>†</sup> Average.

**Example 2:** The stabilizing effects of 2,2,4-trimethyl-6-ethoxy-1,2-dihydroquinoline and salts thereof were studied on carotene in mixtures of corn oil and alfalfa stored in cotton bags at room temperatures (23° to 26°C.) for two months. The following observations were made:

| Wt. percent in mix |                                     | Percent Carotene Retained |              |
|--------------------|-------------------------------------|---------------------------|--------------|
|                    |                                     | After 1 mo.               | After 2 mos. |
| None               |                                     |                           |              |
| 0.010              | Free base                           | 50.3                      | 35.3         |
| 0.016              | HCl salt                            | 71.8                      | 50.8         |
| 0.0147             | H <sub>2</sub> SO <sub>4</sub> salt | 70.1                      | 50.8         |
|                    |                                     | 74.8                      | 60.4         |



## MINERALS AND VITAMINS

Vitamins and minerals are an integral part of a balanced animal food. These processes discuss mineral and vitamin supplements.

### MINERALS

#### Phosphorus and Calcium in Proper Ratio

It is important that animal feeds contain phosphorus and calcium in readily assimilable form, and that the ratio of Ca/P shall be calculated to produce balanced use of those elements by the animal. In the process of P.S.J. Locuratolo (U.S. Patent 3,011,891; December 5, 1961; assigned to Compagnie de Saint-Gobain, France) this is accomplished, generally speaking, by introducing the phosphorus into the feed at least partially in the form of alkali metal polyphosphate or calcium polyphosphate. Thus, the sodium tripolyphosphate is exemplary of useful compounds.

The alkali polyphosphates and the calcium polyphosphates are perfectly assimilable by the animal organism and are compatible with all other ingredients of a good feed, such as vitamins. They have the special advantage of being agglomerable in a solid body in the form of tablets, briquettes, grains, etc., but sufficiently friable to be readily crushed and absorbed by animals without previous dissolution in water. The following are examples of tablets for cattle feed.

#### Example 1:

|                               | <u>Parts by Weight</u> |
|-------------------------------|------------------------|
| Anhydrous dicalcium phosphate | 48.0                   |
| Sodium chloride               | 37.5                   |
| Magnesium sulfate             | 12.5                   |
| Sodium tripolyphosphate       | 4.0                    |

This formula gives a ratio of Ca to P of 1.

#### Example 2:

|                     | <u>Parts by Weight</u> |
|---------------------|------------------------|
| Dicalcium phosphate | 550                    |
| Sodium chloride     | 245                    |

(continued)

(continued)

|                         | <u>Parts by Weight</u> |
|-------------------------|------------------------|
| Magnesium sulfate       | 120                    |
| Sodium tripolyphosphate | 95                     |

This formula gives a ratio of Ca to P of 0.9.

### Introducing Phosphorus into Water Supply

A process for dispensing phosphorus to the water supply of animals is described by J.C. Eller (U.S. Patent 3,294,543; December 27, 1966; assigned to Dawson Chemical Company, Inc.) The material used is sodium triphosphate, an anhydrous white crystalline material also known as sodium tripolyphosphate and STPP. It has an empirical formula of  $\text{Na}_5\text{P}_3\text{O}_{10}$ , and may exist as a powder or granule. Phosphorus content is about 25% and the pH of a 1% water solution is 9.7. The influence of sodium triphosphate on the pH of drinking water in the range of concern is insignificant.

Sodium triphosphate is shown to be soluble to the extent of about twelve parts in one hundred parts saturated solution at 15°C. which is quite satisfactorily soluble in the desired concentration range for the purpose under discussion. Experience has indicated that range cattle normally would require no more than 0.3% STPP by weight, which is well below the limit of 12%. Sodium triphosphate in a powdered or granulated form fully meets the requirements for dry dispensing into drinking water. It is readily soluble in water, but non-hygroscopic. Thus, it can be stored for relatively long periods of time without losing its ability to flow freely. Although it can be introduced into a water tank by hand at proper intervals, it is particularly suited for use in dry dispensing apparatus in which it will not cake or bridge within the hopper or reservoir.

The unique characteristics of STPP render it ideally suitable for service as a vehicle to introduce other additives into the drinking water. The salts of a number of metals which may be needed in animal diets such as manganese, copper, iron, cobalt and zinc are hygroscopic, and thus would be unsuited for dry dispensing. However, when dispersed through sodium tripolyphosphate in the required amounts, these metallic salts will form a very small part of the total bulk of the dry additive material, and their affinity for moisture will present no problem. Similarly, antibiotics such as aureomycin and certain systemic insecticides, all of which are hygroscopic, can be mixed with the phosphorus bearing material for introduction into the diet of the animals.

Based upon the amount of phosphorus present in sodium triphosphate, such material should be added to the drinking water for cattle in the range of from one to three ounces, dry weight, for each eight gallons of water in the drinking tank, or for each animal for each day. The proper amount will depend not only on the amount of phosphorus which may be obtained by the animals in question from the pasture in which they normally feed and similar factors, but also on the condition of the animals themselves. For example, dry cows on pasture under normal rainfall conditions probably would require the minimum amount of one ounce per day. For weaned calves, yearlings, heifers and mature bred cows during the winter season, the amounts might be increased to one and one-half ounces. For cows which are nursing, bulls, fattened yearlings and two year olds, and all cattle on pasture



during low rainfall periods, the maximum amount of three ounces might be recommended. It is to be understood that these figures relate to cattle, and the requirements of other animals can readily be determined based on the factors mentioned previously.

## Carbamide Phosphate

B. Garre (U.S. Patent 3,117,867; January 14, 1964; assigned to Gebruder Giulini GmbH, Germany) found, that a particularly superior livestock feed may be provided by adding to a nutrient feed an effective amount of carbamide phosphate. The amount of carbamide phosphate may range from 0.1 to 1.5% by weight based on the weight of the feed. The carbamide phosphate or urea phosphate contemplated may be expressed by the formula  $\text{CO}(\text{NH}_2)_2 \cdot \text{H}_3\text{PO}_4$ .

In a test study on rats, upon administering to the animals the 0.5 to 1% carbamide phosphate with the feed, the number of sterile females was considerably decreased, and a weight increase per gram of feed consumed was obtained. Thus, a more effective and efficient feed utilization by the animals is attained when the carbamide phosphate in accordance with this process is administered to the animals with the feed. It has also been found that animal feeds which contain phosphate or to which orthophosphates are added in the known manner, undergo an improvement in utilization by the livestock or animal where the feed contains condensed phosphates in admixture with carbamide phosphate. This compensates for any deficiency in the normal growth requirement of resorbable phosphorus in livestock fed with the feed.

In fact, a synergistic effect takes place when both the condensed phosphate and carbamide phosphate are used together, such that the livestock or animals effectively utilize their food so as to increase their rate of growth and also to prevent sterility. It may be considered that the condensed phosphates serve to disperse the carbamide phosphate whereby the carbamide phosphate becomes better distributed and therefore better utilized in the animal organism. Also, the carbamide phosphate may be better retained by adhesion with respect to the feed used.

Conveniently, in accordance with this process a mixture of carbamide phosphate and condensed phosphates, such as alkaline polyphosphates, including tetrasodiumphosphate ( $\text{Na}_4\text{P}_2\text{O}_7$ ) may be added to the feed in an amount of from 0.03 to 2% by weight based on the weight of the feed. In this mixture the amount of carbamide phosphate present is preferably at least 10% by weight of said mixture, this amount being at least 0.1% by weight based on the weight of the feed. Mixtures which may be used in accordance with the process in this regard are set forth below.

1

52%  $\text{Na}_4\text{P}_2\text{O}_7$   
 24%  $\text{Na}_5\text{P}_3\text{O}_{10}$   
 8% Tetrapolyphosphate  
 7%  $\text{KPO}_3$   
 9%  $\text{CO}(\text{NH}_2)_2 \cdot \text{H}_3\text{PO}_4$   
 cp. = 2.0  
 pH = 8.6

2

72.3%  $\text{Na}_4\text{P}_2\text{O}_7$   
 9.0%  $\text{KPO}_3$   
 8.0%  $\text{CO}(\text{NH}_2)_2 \cdot \text{H}_3\text{PO}_4$   
 10.7%  $\text{Na}_3\text{PO}_4$   
  
 cp. = 2.0  
 pH = 9.2

Calcium Orthophosphate Containing Trace Elements

The process of M.G. Gillis (U.S. Patent 2,973,265; February 28, 1961; assigned to International Minerals & Chemical Corporation) provides an improved method of preparing a livestock and poultry feed supplement comprised of a solid calcium orthophosphate and trace elements uniformly distributed therein.

In the preparation of the described livestock and poultry feed supplement, small amounts of compounds of cobalt, copper, iron, manganese and zinc are reacted with an aqueous solution of phosphoric acid. A calcium compound is added to the solution to precipitate a solid calcium orthophosphate material having compounds of cobalt, copper, iron, manganese and zinc uniformly distributed throughout the solid. If desired, a portion of the calcium compound may be replaced by a material selected from the group consisting of ammonia, ammonium compounds, sodium compounds, and potassium compounds.

An aqueous solution of phosphoric acid having an  $\text{H}_3\text{PO}_4$  concentration of between 40% and 65% by weight is used which should contain less than about one-hundredth part by weight of fluorine per part of elemental phosphorus since a fluorine content in excess of this amount may have a toxic effect when fed to livestock and poultry. Trace elements are added in suitable amounts so that the calcium phosphate so formed contains preferably 25 to 100 ppm cobalt, 2,000 to 10,000 ppm copper, 10,000 to 50,000 ppm iron, 5,000 to 30,000 ppm manganese and 10,000 to 40,000 ppm zinc per million parts of phosphorus in the acid solution.

A calcium containing compound is added to the acid in an amount sufficient to provide a  $\text{CaO}/\text{P}_2\text{O}_5$  mol ratio of between 0.9 and 3.0 and preferably between 1.5 and 2.5:1. It is preferred that the particle size of the calcium compound be less than 100 mesh in order that a rapid reaction of the calcium compound with the solution may be obtained.

Example: About 1000 parts of an aqueous solution of phosphoric acid containing about 50%  $\text{H}_3\text{PO}_4$ , was placed in a suitable container and agitated by a mechanical stirrer. Compounds of trace elements in solid form were added to the phosphoric acid in the following amounts:

| Compound                                  | Parts |
|---|-------|
| $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ | 0.56  |
| $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ | 0.54  |
| $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ | 6.80  |
| $\text{MnSO}_4 \cdot \text{H}_2\text{O}$  | 7.93  |
| $\text{ZnCl}_2$                           | 7.70  |

After dissolution of the solids, about 510 parts of calcium carbonate were slowly added to the solution. The viscous slurry which was produced was placed in a ceramic tray and dried in an oven at a temperature of about  $110^\circ\text{C}$ . for a period of about 72 hours. About 721 parts of solid product were obtained. This material was comminuted to a product size less than about 50 mesh. Presented on the following page is a chemical analysis of the feed supplement.



## Minerals and Vitamins

| <u>Element</u> | <u>Percent by Weight</u> |
|----------------|--------------------------|
| P              | 22.7                     |
| Ca             | 29.5                     |
| Co             | 0.019                    |
| Cu             | 0.019                    |
| Fe             | 0.190                    |
| Mn             | 0.358                    |
| Zn             | 0.499                    |

Rats fed on a milk diet supplemented by 2% of the above prepared material showed substantial increases in weight and hemoglobin levels.

### Zinc Oxide Feed Supplement

Zinc oxide is a preferred material in supplying zinc to the animal body inasmuch as zinc oxide is easily soluble in digestive juices and thus readily available to body tissues. Heretofore, difficulty has been experienced in mixing zinc oxide with other minerals and animal feed, due to the fact that zinc oxide is a very light, flocculent, extremely fine material that agglomerates easily.

The process of C.S. Webb (U.S. Patent 2,999,752; September 12, 1961) provides a new zinc oxide material composed of discrete particles which mix readily with the other ingredients of animal feeds and fertilizers. He found that a uniformly mixed composition consisting essentially of zinc oxide and dolomitic lime in specific proportions, and preferably consisting essentially from 87% to 92% by weight zinc oxide, when ground to a particle size in the range of 44 to 100 microns differs sharply from pure zinc oxide in its physical characteristics and in its effectiveness.

The new composition is conveniently manufactured from a zinc bearing ore, such as, for example zinc sulfide, zinc carbonate and the like, such ore being carefully chosen to avoid the presence of undesirable or poisonous heavy metals such as lead, bismuth, arsenic and the like. The zinciferous ore is calcined at a high temperature in an oxidizing atmosphere to eliminate chemically combined elements and to convert the ore to dead-burnt zinc oxide. This product contains an average of approximately 90% zinc oxide which is combined with dolomitic lime and other materials, thereby constituting a highly concentrated nutritional zinc additive composition.

The calcined material is suitably ground by an attrition mill or other like pulverizing apparatus to a fineness of from 44 microns to 100 microns. This reduction in size to the range indicated is a critical limitation. It has been found that when the zinc oxide composition is ground to the size range of from 44 to 100 microns, mixing times are reduced considerably and the fertilizer or feed mix is uniform. When the particle size range increases above 100 microns, the material is too granular and too large for uniform distribution with other minerals. When the particle size goes considerably below 44 microns, the material is characterized by its flocculent nature and tendency to agglomerate.

The finely ground composition is now suited for mixture with other ingredients and chemical compounds constituting the bulk of animal feeds and fertilizers. It is preferably added to

these feeds and fertilizers in such amounts as to constitute by weight on a metallic zinc basis from 0.01% to 1.0% of the total weight of the mixture. The final mixture of the animal feed or fertilizer as used contains extremely small amounts of homogeneously dispersed zinc oxide.

### Iron-Whey Food Supplement

J.R. Simmons and A. Rapport (U.S. Patent 3,421,897; January 14, 1969; assigned to Simmons Milk Products, Inc.) found that when condensed whey is intimately mixed with an iron-containing solid absorbent carrier, such as vermiculite, and the mixture is permitted to cure, the whey on the carrier in the resulting product contains ferric iron in a usable form, possibly a soluble form such as ferric lactate, which may be digested and utilized by the animal.

Example: A condensed and cultured whey can be prepared by recovering whey, having an acidity of about 0.55%, from a cheese vat at a temperature of about 100°F. The recovered whey is delivered to culture tanks for culturing at about atmospheric temperature for about 12 hours, resulting in raising the acidity to about 1%. The resulting cultured whey is pumped through a preheater at a temperature of about 190°F. and is held in the preheater for about 30 seconds. The whey is then charged to an evaporator supply tank. From the supply tank, the cultured whey is conducted to an evaporator to condense the whey to about 62% solids under high vacuum. The evaporation is carried out in a multiple-effect evaporator, and the last stage or effect is maintained at a temperature not exceeding 120°F. The resulting condensed cultured whey can be cooled for use as a feed in this method. It analyzed as follows:

|                    | <u>Percent</u> |
|--------------------|----------------|
| Lactose            | 38             |
| Lactic acid        | 10             |
| Crude protein      | 7              |
| Ash                | 7              |
| Crude fat, minimum | 1/2            |
| Water              | 37 1/2         |

75 lbs. of the above prepared and analyzed condensed cultured whey were mixed with 25 lbs. of Verxite in a rubber-tipped blade mixer at atmospheric temperature until an intimate mixture was obtained. The resulting mixture was placed in a curing tank for 24 hrs. at room temperature. Thereafter, the cured mixture was processed through a feed-finisher, consisting of a hammer mill with stationary hammers, to break the material into divided particles of the desired size. The resulting free-flowing particulate food supplement material was packaged in paper bags having plastic liners. It contained 0.6% soluble ferric iron.

### Chelated Metals in Feedstuffs

B.P. Cardon (U.S. Patent 2,960,406; November 15, 1960; assigned to Erly-Fat Livestock Feed Co.) discovered that, when metal deficiencies are made up by the use of chelated metals, bacteria development and growth are so greatly enhanced by the chelate form of the make-up metals that digestibility often increases to as much as 75%, and bacteria activity appears to go beyond normal. Some of these metals are taken up by the bacteria



to activate and vastly increase their numbers, which also increases fermentation and subsequent digestion in the intestines. These bacteria increases themselves provide an additional nourishment factor for the ruminant animal, when they pass on into the intestines for normal digestion and absorption. Such absorption includes not only the metal constituents taken up by the bacteria but also excess metal constituents over the requirements of the bacteria. Thus, when metal deficiencies are corrected with chelated metals, bacteria activity and digestibility of cotton gin waste roughage and other waste roughage, go well beyond normal.

More specifically, digestibility may be increased in the presence of chelated nutritional metals, as described, to as much as around 5% to perhaps 15% above normal. These improvements due to the presence of chelated trace metals, added to the improvements produced by the presence of a good bacteria food additive, such as molasses, make ruminant feeding with otherwise low-digestibility cellulosic waste a very important phase of beef and dairy cattle feeding. In addition, the supplying of chelated nutritional metals to feeds for simple-stomached animals, such as poultry and swine, is of much importance in areas of mineral deficiencies.

These metals must be in chelated form to meet the requirements of adequate solubility and availability both from the standpoint of need therefore by the bacteria and from the standpoint of adequate use and absorption in the digestive system of the animal. As an example of an additive to a feedstuff to meet mineral deficiencies in a particular beef cattle area, the following table is supplied as representing an approach to make up deficiencies in cobalt, copper, manganese, iron, potassium, and zinc, the chelating agent being any non-toxic acceptable agent, preferably ethylene diamine tetraacetic acid. Such additive is:

|                                      | <u>Lbs.</u> |
|--------------------------------------|-------------|
| CoSO <sub>4</sub> ·7H <sub>2</sub> O | 4.0         |
| CuSO <sub>4</sub> ·5H <sub>2</sub> O | 5.0         |
| MnSO <sub>4</sub> ·H <sub>2</sub> O  | 6.0         |
| FeSO <sub>4</sub> ·7H <sub>2</sub> O | 7.0         |
| ZnSO <sub>4</sub> ·H <sub>2</sub> O  | 3.0         |
| Chelating agent                      | 74.0        |
|                                      | <hr/> 99.0  |

For a given feedstuff requiring all of the above constituents to make up deficiencies in a substantial area, one pound of the above chelating mixture was dissolved in 2.5 gallons of water, and such solution was thoroughly distributed in 2 1/2 tons of a cattle feed. This amounted to supplying 0.4 pound of the chelated metal mixture per ton of feed. Since the amounts of trace metals to be supplied are not severely critical, the chelated metals may be added to the amount of about 1/2 pound per ton. Where an iodine deficiency exists, iodine may be included, such as about 1% of KI in the above additive composition. Similarly, magnesium calcium and phosphorus may be included in appropriate form and amount usable for animal feeding. The range of individual metallic constituents may be varied more or less to meet deficiencies.



Feed Containing Magnetically Attractable Iron

C.G. Harrel and W.A. Bosin (U.S. Patent 2,928,739; March 15, 1960; assigned to The Pillsbury Company) have developed a process which provides feed materials with an edible iron nutrient compound so selected as to be capable of substantially total magnetic extraction.

A method of testing is provided which, within the bounds of experimental error, will quickly and efficiently extract substantially all of the nutrient iron fraction in a random sample of feed. By comparing with known feeds and concentrates containing preselected amounts of nutrient iron in magnetically separable form, such testing method will serve to detect the thoroughness of mixing of the feed material and may be used to identify the type and source of feed or feed concentrate by microscopic examination of the configuration of the preselected iron particles and the relative proportions thereof.

Figure 8.1a is a schematic representation of the steps in the process of making the concentrate and mixed feed material and the method of testing; Figure 8.1b is a perspective view of the magnetic element utilized for test purposes, a portion of the element being cut away in vertical section to show the construction thereof; Figure 8.1c is a magnified view of sized carbonyl iron particles employed as tracer material; Figure 8.1d is a similar magnified view of sized iron particles of the ferrum reductum type; and Figure 8.1e is a magnified view of iron recovered from a random test sample showing the approximate relative distribution between the carbonyl iron and the ferrum reductum.

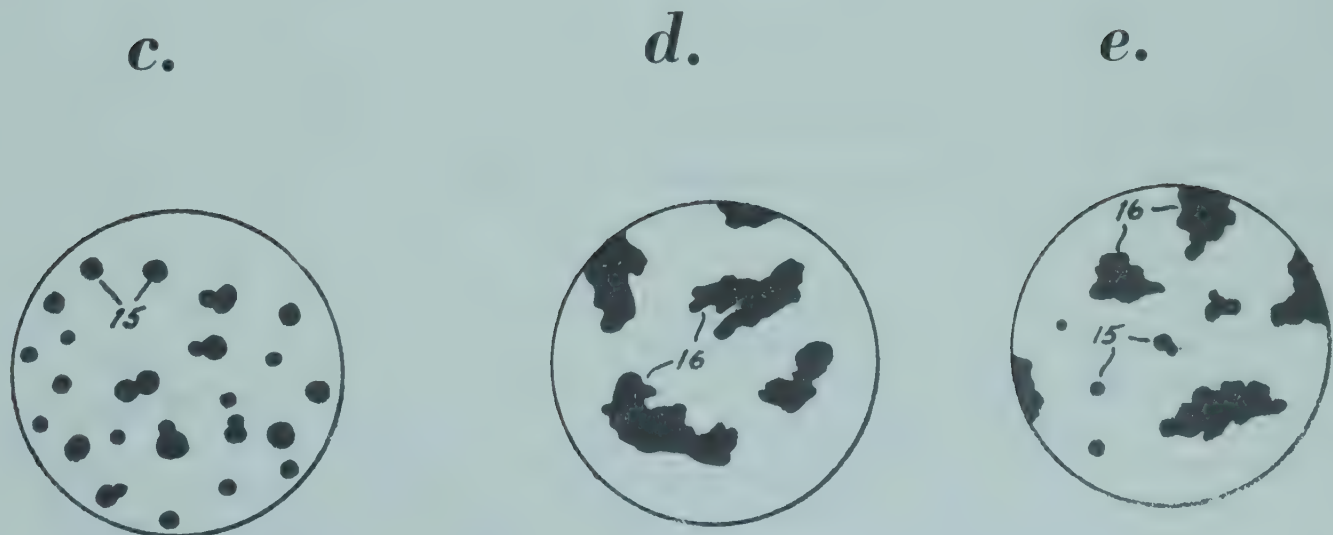
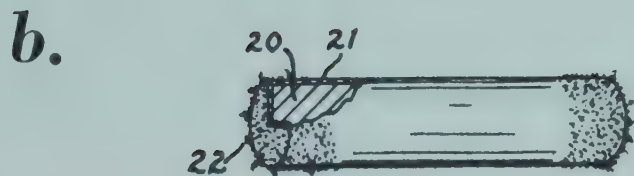
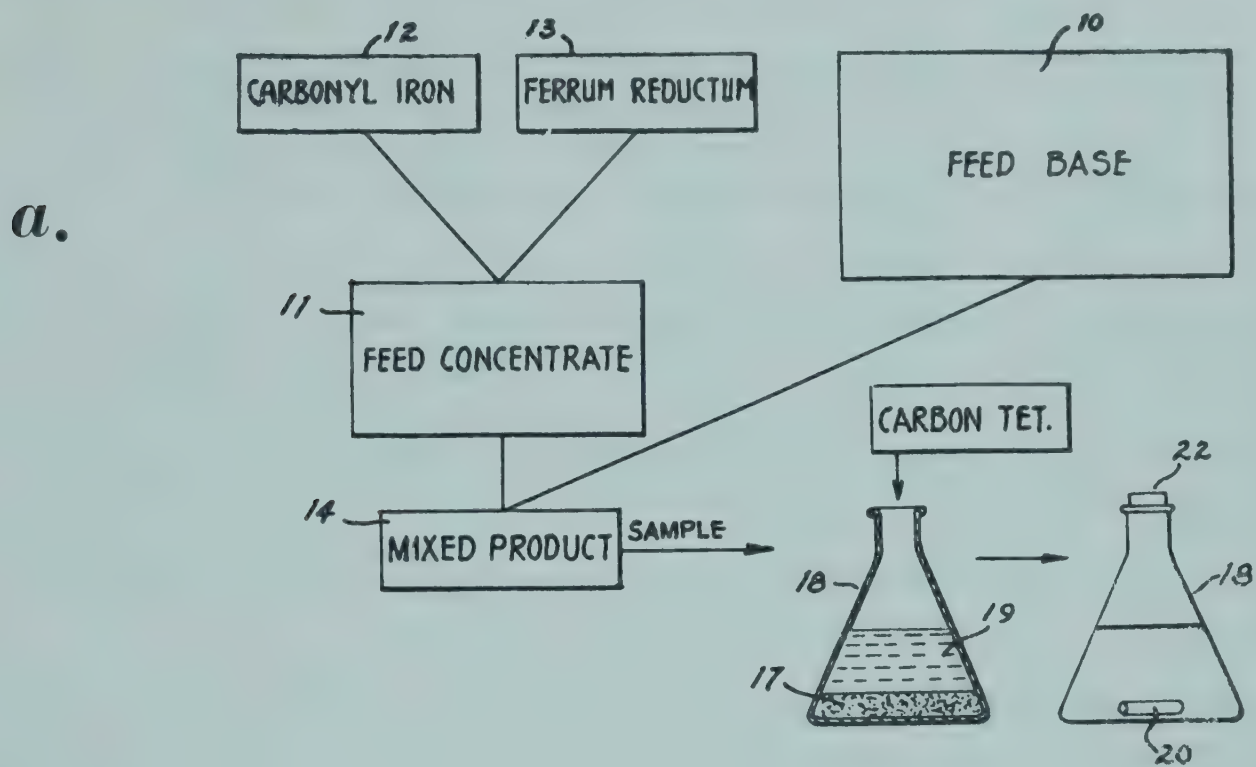
In Figure 8.1a, the general mixing procedure is outlined. The feed base is indicated by box 10 and may comprise any general ground product such as ordinary mash which is improved by the addition of a feed fortifier or concentrate 11. In the usual course of events, the iron component designated as 12, 13, or both, will be intermixed with the feed concentrate 11 which usually contains other minerals and vitamins as well as cereals. The concentrate is then added to the feed base in recommended proportions and thoroughly intermixed to produce the mixed product 14. As pointed out, the small quantities of at least some of the concentrate materials make it mandatory that they be properly mixed throughout the entire mass of the feed base. If it is desired to use the iron component as a tracer to determine the thoroughness with which the concentrate has been mixed, then it is incorporated therewith at the time the concentrate is compounded.

If it is merely desired to determine the dispersion of the components in the ultimate mixed product, then the iron may be added to the concentrate and the feed base at the time they are first brought together. In either case, random sampling at several locations in the mixed product which demonstrates uniformity as to the amount of iron present is proof of thorough intermixing of all the components.

In order to effect magnetic separation of the iron for test purposes, a magnetically attractable form is used. It has been found that either the form known as carbonyl iron or that known as ferrum reductum serves admirably for the purposes herein disclosed. For coding purposes, mixtures of both may be employed. Referring to Figure 8.1c, the configuration of carbonyl iron is shown as it appears under the microscope. The iron particles are spherical and easily distinguishable as such when examined under magnification. Referring



FIGURE 8.1: FEED CONTAINING MAGNETICALLY ATTRACTABLE IRON



Source: C.G. Harrel and W.A. Bosin; U.S. Patent 2,928,739; March 15, 1960

to Figure 8.1d, the configuration of ferrum reductum appears. The latter is characterized by nondescript or random configuration which seldom includes anything which can even be mistaken for a spherical shape. The individual particles 15 of the carbonyl iron and the individual particles 16 of the ferrum reductum are useful for the purposes herein disclosed when lying within the size range of average diameters including three to twenty microns. Iron particles within the range of diameters noted are not only easily digestible by animals but also are capable of magnetic separation as will be subsequently disclosed.

Referring again to Figure 8.1a, a series of random samples are taken from the mixed product to determine the uniformity of mix at various locations therein. Where it is desired to test the uniformity of mix in the feed concentrate, the same procedure may be employed. For test purposes, 100 gram samples indicated at 17 are placed in a flask 18 and 175 milliliters of fluid, indicated at 19, are placed in the flask 18 to freely suspend the individual particles. Any noncorrosive fluid which will not thicken or react with the mixed product may be utilized for this purpose. Carbon tetrachloride is found to be satisfactory and, because of its solvent quality, insures against oils and other sticky substances becoming adhered to the iron particles.

Into the suspended product is placed a prepared magnet 20 which must be capable of maintaining uniform weight and easily cleaned between usages. To this end, it has been found that a thin coating 21 of plastic material such as Teflon is useful and will not interfere with the magnetic properties of the magnet 20. An enlarged view of the magnet is shown in Figure 8.1b. In preparing the magnet, it is first rinsed with carbon tetrachloride, then dried at 100°C. for ten minutes. The magnet is weighed on an analytical balance and then placed in the flask 18 as previously noted. A stopper 22 is then applied to the flask and it is swirled in inverted position for at least thirty seconds. The flask is then righted and swirled again for another thirty second interval.

This procedure is repeated for several times to effect intimate contact between the magnet 20 and all of the particles of the mixed product 17. The mixture is then poured from the flask 18. The plastic coated magnet and its adhering iron is then washed with carbon tetrachloride to remove adhered mash and fat particles. The magnet and its adhered iron particles are then laid on a watchglass and dried in an oven at 100°C. for 10 minutes. The magnet and iron particles are then lightly brushed with a camel's hair brush to remove any dried feed particles which may have become entrapped by the magnetically clinging iron particles 22 which cluster on the magnet, as shown in Figure 8.1b.

The magnet and its adhering iron are then weighed and the difference in weight between the latter and the magnet taken alone gives the weight of iron removed from the sample. When it is desired to make another test, the iron particles 22 are wiped from the magnet 20 and the procedure is repeated. Since the feed base 10 may itself have small traces of magnetically attractable iron, a blank sample is taken at several locations prior to admixing the concentrate and iron component. An average may be made of the blank runs and the trace of iron found therein is then deducted from the amount of total iron collected by the magnet 20.

In the following series of tests, the mixed product constitutes a 40% feed concentrate and a 60% corn meal feed base.



## Minerals and Vitamins

| <u>Sample</u> | <u>Parts Per Million of Iron in Sample</u> | <u>Weight of Mixed Product Used, g.</u> | <u>Weight of Iron Added</u> | <u>Weight of Iron Recovered</u> | <u>Iron Recovered Iron Added</u> | <u>Percent Iron Recovery</u> |
|---------------|--|---|-----------------------------|---------------------------------|----------------------------------|------------------------------|
| 1             | Blank                                      | 100                                     | None                        | 0.0014                          | 0.0014                           | - -                          |
| 2             | 50   | 100                                     | 0.0062                      | 0.0076                          | 0.0014                           | 100.0                        |
| 3             | 100  | 100                                     | 0.0110                      | 0.0133                          | 0.0023                           | 108.2                        |
| 4             | 200  | 100                                     | 0.0206                      | 0.0215                          | 0.0009                           | 97.6                         |
| 5             | 200  | 100                                     | 0.0222                      | 0.0228                          | 0.0006                           | 96.4                         |
| 6             | 300  | 100                                     | 0.0294                      | 0.0317                          | 0.0023                           | 103.1                        |

### Granular Mineral Feed Supplement

C.C. Jensen and F.X. Doody (U.S. Patent 3,464,824; September 2, 1969; assigned to Darling & Company) describes a process for developing a granular feed supplement.

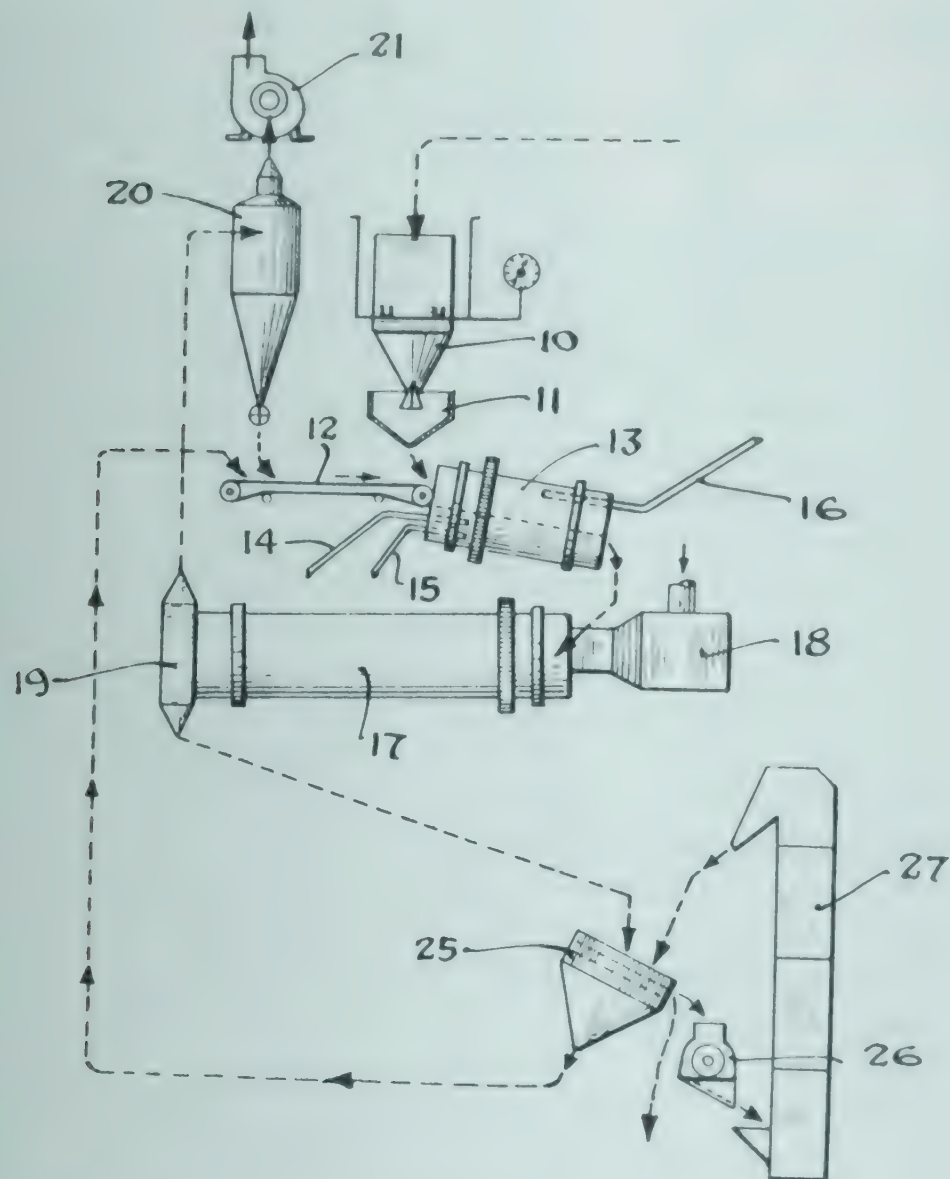
#### Example:

|                               | <u>Pounds</u> |
|-------------------------------|---------------|
| Soft phosphate                | 925           |
| Limestone (calcium carbonate) | 852.5         |
| Salt (sodium chloride)        | 180           |
| Trace mineral premix          | 12.5          |
| Steamed bone meal             | 5             |
| Dicalcium phosphate           | 5             |
| Iron oxide                    | <u>20</u>     |
|                               | 2000          |

The above mineral feed ingredients are weighed and mixed in a weighing hopper 10 (see Figure 8.2), screened to exclude foreign materials and held in a surge hopper 11 from which the premixed ingredients are continuously charged by means of an endless conveyor belt 12 into a continuously rotating inclined open cylindrical agglomeration shell 13. The cylindrical shell 13 is about 17 ft. long and 6 1/2 ft. in diameter, has a smooth inner wall surface without baffles, and is rotated about its longitudinal axis at a rate of about 9 to 11 rpm.

The rotating shell 13 is provided at its upper or charging end with a water sparger pipe 14 and a steam sparger pipe 15 disposed within the shell adjacent the lower surface thereof below the normal level of the bed of mineral feed materials. Water at a temperature of about 140°F. and steam at a temperature of about 212°F. are each introduced through the sparger pipes 14, 15 at a rate of about 100 to 200 pounds per ton of mineral feed ingredients. The water and steam dissolve the water soluble mineral feed ingredient, which in the preferred embodiment is comprised mainly of sodium chloride, and the dissolved soluble feed ingredient uniformly mixes with and moistens the finely divided particles of essentially water insoluble mineral feed ingredients. As the moistened particles are tumbled in the rotating

FIGURE 8.2: GRANULAR MINERAL FEED SUPPLEMENT



Source: C.C. Jensen and F.X. Doody; U.S. Patent 3,464,824; September 2, 1969

cylindrical shell 13 the particles agglomerate and form larger particles while the mineral feed materials advance longitudinally through the rotating shell 13. Adjacent the discharge end of the rotating shell fine water sprays 16 are provided above the bed of material to facilitate finally adjusting the moisture content and the condition of the agglomerated mineral feed material immediately before discharge from the rotating shell.

When operating under the foregoing conditions agglomerated mineral feed material having a temperature of about 175°F. is discharged from the rotating shell at a rate of about one ton every 10 minutes. The rate may vary from about one ton every 6 minutes to one ton every 12 minutes, depending on whether the ambient temperature is above normal or below normal. The water and steam additions are continuously regulated under preferred operating conditions so that the agglomerated material discharged from the rotating shell has a particle



size below about 2 mesh and above 20 mesh. The agglomerated material discharged from the rotating agglomeration shell is charged directly into a rotating inclined cylindrical dryer 17 about 40 ft. long and 7 ft. in diameter with a gas fired combustion chamber 18 disposed at the inlet end thereof. The combustion chamber 18 heats air to be drawn through the dryer 17 at a temperature of about 300°F. The lower end of the cylindrical dryer 17 is provided with an exit stack 19 through which drying gases pass upwardly to a cyclone-type dust collector 20 having a blower 21 associated therewith for drawing the drying gases through the cylindrical dryer and dust collector.

The combustion chamber which heats the hot air for drying the agglomerates is controlled by the temperature of the exit stack 19 and preferably is maintained between 140° and 200°F., with the optimum stack temperature being between 185° and 200°F. The rotating cylindrical dryer 17 having a diameter of 7 ft. preferably has about 20,000 cubic feet per minute of hot air drawn therethrough while the dryer 17 is rotated at a rate of about 8 rpm. During the passage of the agglomerates through the dryer 17 the moisture content is reduced to between 2.4% and 5.1% by weight and preferably to about 3.5% and 4% by weight. The gas leaving the rotating dryer containing entrained fine dust passes through the cyclone dust collector 20 wherein the dust particles are removed, and the fines are deposited on the charging conveyor belt 12 and recharged to the rotating agglomeration shell 13.

The product size of the granular feed material is preferably controlled by passing the discharge from the cylindrical dryer 17 to a double deck electrically vibrated screen 25. The mineral feed granules having a size greater than 8 mesh are ground in a grinding mill 26 and returned by elevator 27 for rescreening on the double deck screen 25, while the granules having a size less than 16 mesh are recycled and charged to the rotating agglomeration shell 13.

The granular mineral feed product as above produced has a particle size distribution between 8 and 16 mesh and has an approximate analysis of between 16 and 19% calcium, about 4% phosphorus, between 8 and 10% sodium chloride, and about 0.008% iodine, all on a dry weight basis. The granular mineral feed product has a density of about 50 to 60 pounds per cubic foot, requires an average compression strength of 0.31 pound to crush granules thereof having a mean diameter of 2.19 mm., and exhibits substantial resistance to crushing or dusting during normal handling. The mineral feed granules produced were capable of being shipped in conventional bags and admixed with conventional feed materials without disintegrating appreciably or forming an objectionable amount of dust so that substantial savings are possible for the feed mill operator and the livestock owners because of the substantially dustless properties of the granular mineral feed product of this process.

The feed granules prepared in the foregoing manner are used by mixing with the normal rations for hogs, beef cattle, dairy cattle, calves, sheep, broiler, starter, growing or turkey mashes in a ratio of 2.5 pounds mineral feed granules to each 100 pounds of normal ration or one 50-pound bag of feed granules per ton of ration. In laying mash, it is preferred to use 5 pounds of mineral feed granules to each 100 pounds of ration or two 50-pound bags per ton of ration.

While "soft phosphate" has been used in the foregoing example, other sources of feed grade phosphate or phosphorus compounds characterized by their ability to provide an available



source of phosphate and exhibit a low fluorine content can be used in accordance with approved nutritional practice. Among the materials which can be used in finely divided form are bone meal, bone black, dicalcium phosphate, defluorinated phosphate, defluorinated super phosphate, raw rock phosphate, phosphate, Curacao, and phosphatic limestone. In the formulation other sources of calcium instead of limestone can also be used, including oyster shell flour, calcite, gypsum, and wood ashes, all in a very finely divided form. It should also be understood that the feed granule composition can be modified to include other ingredients, such as vitamins or antibiotics, as desired.

### FAT SOLUBLE VITAMINS

#### Stable Vitamin A Premix

P. Iacono and S.M. Weisberg (U.S. Patent 2,933,392; April 19, 1960; assigned to National Dairy Products Corporation) has a process for preparing a fat-soluble vitamin composition of high stability at elevated temperatures approximating 100° to 110°F. (37.8° to 43°C.) is provided which includes an edible fatty material, an antioxidant, lecithin, milk solids (including milk protein) and a fat-soluble vitamin. Vitamin A is a preferred vitamin, but instead of or in addition thereto, the composition may contain other vitamins and ingredients which do not affect the stability thereof, such as the D, K and E vitamins.

Example: Skim milk was condensed to a solids content of 23% solids (758 lbs.) and brought to 140°F. with constant agitation and held at that temperature for 20 minutes to pasteurize it. The milk then was cooled to 100°F. Coconut oil (70 lbs.) was melted and with the aid of a Lightnin stirrer there was blended therein 681 g. of Yelkin (soya lecithin), 112.3 g. Tenox II (a mixture of butylated hydroxyanisole, propyl gallate and citric acid dissolved in propylene glycol) 920 g. of vitamin A palmitate (1.36 million units per g.) and 497.2 g. of vitamin D (1 million units per g.). The mixture then was added to the pasteurized skim milk and the batch homogenized at 3,000 psi and 120°F. The pH of the mixture was 6.25, and the solids content 30%.

The batch was fed into a spray-drier at a rate of 4 gallons per hour, an inlet air temperature of 300°F. and an outlet air temperature of 185°F. under a pressure of 2,000 psi. The composition of the powder ranged from 97.7% to 98.2% solids and included: fat 27.9 to 31.5%, protein 24.8 to 23.6%, lactose 40.7 to 37.2%, ash 6.24 to 5.77%, acidity as lactic acid 0.59 to 0.60%, vitamin A 10,847 to 11,570 IU per gram. The stability of the preparation was determined by an accelerated storage test in which a sample of the material was held for 500 hours at 110°F. in air. At the conclusion of the test the vitamin A value was 10,595 to 10,202 IU per gram. This is indicative of an extraordinary vitamin A stability, since the test represents severe oxidative conditions. This material is useful as a premix for a dry animal feed formulation.

The dispersibility tests show that the vitamin A powder was considerably more readily dispersed than the whole milk powders which had been prepared under the same conditions as the vitamin A powder. The photo turbidity measurements show that reconstitutability at very low dispersing energies was as good as the whole milk powders.



### Stabilized Fat-Soluble Vitamins

A. Rosenberg (U.S. Patent 2,937,091; May 17, 1960) discovered that it was possible to stabilize fat-soluble vitamins by the creation of dry, discrete spheres or beads of small size, i.e., of about 2 mm. or less in diameter, in which the vitamin is a component of a central core of fat that is encased within an outer protective shell. The spheres or beads are manufactured by dissolving a vitamin bearing oil or fat-soluble vitamin in the basic molten fat; adding (1) this vitamin-containing fat to (2) an aqueous solution of the proteinaceous material; heating the mixture of (1) + (2) to a temperature at which the protein could coagulate if the pH were more favorable; homogenizing the mixture to form an oil in water emulsion and spray-drying the emulsion. The dry vitamin spheres are free flowing, uniform and similar in structure, and will not "oil off" under regular conditions of storage for indefinite periods. Fats that are solid at room temperature, and especially those that remain solid at room temperature, after blending with the vitamin oils are preferred.

The proteinaceous material used should contain on the dry basis at least 20% of protein and should be heat coagulable in aqueous solution at a temperature of 65° to 85°C. when the pH of the solution is between 4.0 and 5.0. Proteinaceous material that has already been denatured by heat-processing so that the material is no longer soluble in water, cannot be used in making the products of this process. Solvent-extracted but not heat-processed soy flour is preferred as the material to form the outer protective shell that encases the vitamin-fat cores of the spheres, but other proteinaceous materials such as wheat gluten and low-heat skim milk powder (or skim milk) serve also. The object is to obtain a tough, water-resistant proteinaceous shell or film around the fat core, i.e., one that does not release the fat component simply on the addition of water. The proteolytic enzymes in the digestive tract solubilize the tough protein film, thereby making the vitamin-fat core available for digestion and absorption.

Example: In one tank 97.0 parts of coconut oil, hydrogenated to an iodine number of 1.0 and having a melting point of 37°C., is heated to about 47°C. To this are added 0.02 part of butylated hydroxyanisole, 0.025 part of crystalline vitamin D<sub>3</sub> of a potency of 40,000,000 AOAC units per gram, and 3.0 parts of vitamin A palmitate in corn oil having a potency of 1,100,000 USP units per gram. The mixture is stirred under nitrogen for a period of about 15 minutes to effect a clear solution.

In another tank 31 parts of solvent-extracted non-heat processed soy flour containing about 50% protein (nitrogen x 6.25) are suspended in 300 parts of warm water at about 40°C. and stirred for a period of about 15 minutes until the soy flour is dissolved. Two parts of citric acid are then dissolved in this solution. The contents of the two tanks are mixed under nitrogen and heated by steam coils to a temperature of about 65°C. for a period of 20 minutes. The aqueous-fat mixture is homogenized at a pressure of 3,000 pounds per square inch to yield a fat in water emulsion.

The emulsion containing about 22 to 33% of solids is spray dried as described to produce dry, discrete beads or spheres (hereinafter referred to as the "final product"). This final product has the physical structure of a vitamin fat core encased within a protective spherical coating of the water resistant heat denatured protein film, the diameter of the spheres measuring less than 2 mm. and the spheres containing less than 1% moisture. The ratio of the

## Minerals and Vitamins

fatty vitamin containing core to the protective coating is as 75:25. Assays conducted on the final product as thus manufactured indicated a vitamin A content of 11,000,000 USP units per pound (a loss of only 2% as a result of the process) and 3,400,000 AOAC units of vitamin D<sub>3</sub> per pound (no measurable loss as a result of the process).

When one part of this product is added to 39 parts of the mineral supplement previously described, and this mixture of minerals and the vitamin product is stored at 45°C. for a period of 21 days, fully 88% of the vitamin A and all of the vitamin D<sub>3</sub> are retained, the vitamin A dropping from an initial value of 605 units per gram to 532 units per gram while the vitamin D<sub>3</sub> remained constant at 185 to 190 units per gram. In complete feed mixtures made from this final product, simulating the complete feed mixtures described by Melnick, no measurable losses of the vitamins were noted after the 21-day holding test at 45°C. In the above example, AOAC units of vitamin D<sub>3</sub> are synonymous with International Chick Units.

Vitamin A supplements when stored in a mineral mixture described below at 45°C. at 85% relative humidity show a loss of 50 to 100% of vitamin A in 10 days (equivalent to 3 months at room temperature). The mineral mixture composition is as follows:

|  | <u>Grams</u> |
|--|--------------|
| Manganese sulfate, $\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$ | 74           |
| Potassium iodide, KI   | 6            |
| Ferrous sulfate, $\text{FeSO}_4 \cdot 4\text{H}_2\text{O}$   | 73           |
| Copper sulfate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$    | 11           |
| Zinc sulfate, $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$      | 4            |
| Cobalt sulfate, $\text{CoSO}_4 \cdot \text{H}_2\text{O}$     | 0.6          |
| Calcium carbonate, q.s. 20 pounds.                           |              |

A. Rosenberg (U.S. Patent 2,973,266; February 28, 1961) has found that his bead containing stabilized fat soluble vitamin in a fatty core encased in a heat denatured water resistant proteinaceous digestible shell (see above) is stable under similar conditions.

Example — Stability: When one part of this product is added to 39 parts of the mineral supplement above described, and this mixture of minerals and the vitamin product is stored in a moist environment of about 85% relative humidity at 45°C. for a period of 21 days, fully 85% of the vitamin A and all of the vitamin D<sub>3</sub> are retained, the vitamin A dropping from an initial value of 593 units per gram to 504 units per gram while the vitamin D<sub>3</sub> remained constant at about 187 units per gram. In complete feed mixtures made from this final product no measurable losses of the vitamins were noted after the 21 day holding test at 45°C.

### Colored Fat Soluble Vitamin Particles

M.J. MacMillan and C.M. Ely (U.S. Patent 3,438,781; April 15, 1969; assigned to Diamond Shamrock Corporation) found that when a nontoxic dye is formulated in combination with a fat-soluble vitamin, preferably vitamins A and D, a water-soluble dextrin and a non-toxic edible antioxidant into a product comprising a multiplicity of substantially solid particles, the colored particles thus produced have an enhanced vitamin stability and can easily



be detected in animal feed by the human eye without the need for removing the vitamin-containing particles from the feed or utilizing complicated analytical techniques. The following example is directed to producing colored vitamin A palmitate containing particles having a vitamin potency of about 125,000 USP units per gram.

Example 1: A dextrin solution was prepared by charging under constant agitation 88.62 parts by weight of canary dextrin produced by the pyroconversion of corn starch, said dextrin having a DE of about 3% into 60 cc of water which had been heated to 80°C. The corn starch dextrin quickly dissolved in the water and the temperature of the solution was maintained at 60°C. To this solution there was added 0.50 part by weight of FD &C dye Red #4. The dye dissolved in the dextrin solution at the temperature of 60°C.

A vitamin A palmitate mixture was prepared by mixing 1.41 parts by weight of ethoxyquin and 0.47 part by weight of butylated hydroxy toluene with 9 parts by weight of vitamin A palmitate (1,500,000 units of vitamin A per gram) by agitation under a nitrogen atmosphere at a temperature of 45°C. After a uniform mixture of vitamin A palmitate was obtained, this mixture was added to the dextrin solution while maintaining the temperature at 60°C. The dextrin solution was thoroughly admixed by agitation under a nitrogen atmosphere with the vitamin A palmitate mixture. After about 10 minutes, agitation was stopped and the resulting fluid was homogenized in a Waring blender for about two minutes.

The resulting homogenized material was a smooth, red colored liquid. This liquid was then dried overnight on a stainless steel sheet at about 25°C. The dried material was scraped off the sheet with a spatula in the form of large curled flakes. The flakes were then granulated through a series of USS Wire Mesh Sieves to produce a material which passed through 20 mesh screen but was retained by a 200 mesh screen. Each flake of the granulated material had a bright red color. The product was assayed for its vitamin A palmitate content. The vitamin potency of these particles was about 125,000 USP units per gram. The active particle count per million units of vitamin potency was 2,130,000.

The active particles per million units of vitamin potency of the products was determined in the following manner. The amount of particles in 1 gram were counted. The active particle count per million units of vitamin potency was determined by dividing the known potency per gram of each of these products into 1,000,000 and multiplying this result by the number of active particles visually counted in one gram. The greater amount of particles per unit of vitamin potency of the product insures the production of colored particles which are easily visible when mixed into feeds. Additionally, this high active particle count insures a better uniform dispersion when these particles are mixed into feeds which in turn results in better and more uniform animal nutrition.

Example 2: This example is directed to a visual determination of dextrin-containing vitamin particles in animal feeds. 15 lbs. of a feed was admixed with 0.24 gram of the red dextrin-containing vitamin A palmitate particles prepared in Example 1 (potency of 125,000 USP units of vitamin A per gram) and 0.035 gram of dextrin-containing vitamin D particles (having a vitamin potency of 200,000 USP units per gram). The final feed had a vitamin A potency of 2,000 IU per lb. and a vitamin D potency of 500 IU per lb. The vitamin particles and the feed were thoroughly admixed by means of mechanical agitation. After thorough admixing, the vitamin fortified feed was analyzed.

## Minerals and Vitamins

The dry determination test is carried out in the following manner. One-quarter pound of animal feed which is fortified with the colored vitamins is screened through a 40 mesh sieve. The portion that goes through the 40 mesh sieve is rescreened through a 60 mesh sieve. Approximately 15 grams of the vitamin-containing feed is retained on the 60 mesh sieve. The portion that is retained on the 60 mesh sieve is divided into eight samples and placed on separate circular pieces of No. 4 Whatman White Filter Paper having a diameter of about 10 inches. The paper containing the feed is then visually examined for specks of color. Each observed speck of color is mounted and recorded.

In preparing the wet determination, the paper containing the feed which is utilized for the dry determination is folded over to form a semicircle so that the bottom inside portion of the paper contains the feed. The paper is then completely wetted by means of spraying water over the paper. After the paper is sopping wet, the paper is manually pressed together so that the inside surface of the paper contacts the colored particles. After pressing is completed, the filter paper is opened up to its original shape and the paper is visually scanned for color marks which appeared on the surface of the paper. These color marks are counted and recorded.

### Visual Determination of the Colored Particles Present in the Feed By Means of Wet and Dry Examination

|         | Dry Examination             |                              |                               | Wet Examination         |                          |                           |
|---------|-----------------------------|------------------------------|-------------------------------|-------------------------|--------------------------|---------------------------|
|         | Red<br>Particles<br>Counted | Blue<br>Particles<br>Counted | Total<br>Particles<br>Counted | Red<br>Spots<br>Counted | Blue<br>Spots<br>Counted | Total<br>Spots<br>Counted |
| Sample: |                             |                              |                               |                         |                          |                           |
| 1.....  | 18                          | 23                           | 41                            | 23                      | 24                       | 47                        |
| 2.....  | 19                          | 25                           | 44                            | 23                      | 27                       | 50                        |
| 3.....  | 32                          | 33                           | 65                            | 19                      | 38                       | 57                        |
| 4.....  | 22                          | 24                           | 46                            | 27                      | 23                       | 50                        |
| 5.....  | 27                          | 29                           | 56                            | 33                      | 26                       | 59                        |
| 6.....  | 21                          | 26                           | 47                            | 38                      | 34                       | 72                        |
| 7.....  | 32                          | 30                           | 62                            | 40                      | 33                       | 73                        |
| 8.....  | 37                          | 36                           | 73                            | 32                      | 35                       | 67                        |
| Average | 26.0                        | 28.3                         | 54.3                          | 29.4                    | 30.0                     | 59.4                      |

Colored vitamin particles can be prepared from other fat soluble vitamins (E, D, K). These vitamin containing particles were stable and biologically available.



## GROWTH-PROMOTING CHEMICAL ADDITIVES

Considerable research has been devoted to the development of feeds useful for stimulating growth and improving the feed efficiency of domestic animals. It is known that a wide variety of hormones and antibiotics serve this purpose. This chapter is devoted to active ingredients (which are neither hormones nor antibiotics) but which nevertheless stimulate growth rate and improve feed efficiency.

These active ingredients are divided into two parts. The first section deals with additives which are claimed to have general growth-promoting effects for all animals (i.e., poultry, ruminants, and nonruminants). The second group is devoted to additives showing growth-promoting activity on both swine and poultry. Growth promoters specific only to poultry, ruminants, and swine are reported separately (Chapters 10, 11, and 12).

### GENERAL — POULTRY, RUMINANTS, NONRUMINANTS

#### Auxines

K. Kaemmerer (U.S. Patent 2,925,341; February 16, 1960; assigned to Farbenfabriken Bayer AG) discovered that certain auxines, heteroauxines, or calines constitute a new growth-promoting feed supplement of superior efficiency in its growth-promoting effect. Their use enables the production of larger animals in a shorter time than previously achieved. Additionally, these growth-promoting supplements permit considerable savings of other nutrients in the feed of cattle and other livestock, chickens and other poultry.

The feed supplement contains as its essential active ingredients such auxines, heteroauxines and compounds of the bios-group like the biotin known as vitamin H, which have a specific activity on the multiplication and the growth of the cells of plants. Such compounds are for instance: alpha-naphthyl acetic acid and its salts, 2-methyl-chloro-phenoxy acetic acid and its salts, 2,4-dichloro-phenoxy acetic acid and its salts, 4-phenyl acetic acid and its salts, further beta'-indolyl acetic acid and phenoxy acetic acid and their salts. The amounts of active ingredients contained in animal feed are between 0.0005 and 0.005% calculated on the dry weight of the feed.

Example 1: The addition of the auxines to the animal feed permits to favorably influence the paunch flora of ruminants and to stimulate an increased protein production. This can be established by the fermentation process in the artificial paunch. The following table

## Growth-Promoting Chemical Additives

shows the range of activity of some of the auxines and also shows the increase of the protein formation.

|         | active ingredient                         | range of activity in mg. per 125 ml. | optimum concentration in mg. per 125 ml. | protein formation in percent |               |
|---------|---|--------------------------------------|--|------------------------------|---------------|
|         |   |                                      |  | without growth               | with promoter |
| (1).... | sodium- $\alpha$ -naphthyl acetate.       | 1-60                                 | 20                                       | 100                          | +208          |
| (2).... | sodium-2-methyl-4-chloro-phenoxy acetate. | 1-60                                 | 25                                       | 100                          | +465          |
| (3).... | 2,4-dichlorophenoxy acetic acid.          | 1-40                                 | 20                                       | 100                          | +542          |
| (4).... | sodium phenyl acetate.                    | 1-50                                 | 20                                       | 100                          | +230          |
| (5).... | $\beta$ -indolyl acetic acid.             | 0.1-20                               | 1  | 100                          | +398          |

Example 2: In a poultry feed used for the raising of chickens, small amounts of auxines are added. It is found that the chickens show improved growth response inside the same period of feeding compared with the controls which have been fed without these supplements. At the same time the chickens have a smaller feed consumption. The following table shows the action of the feed supplements in the raising of chickens. The basic feed contains 16.63% of total protein, 2% of animal protein, 70.4% of total nutrients. Group 7 obtained a deficient feed of 16.44% of total proteins without animal protein and a total nutrient amount of 70.6%. The average starting weight of the chickens was 112 grams.

|         | active ingredient                            | percent in feed | average weight increase inside 4 weeks | percent improvement of increase in comparison with No. 7 | percent improvement in feed utilization for 100 grams in comparison with No. 7 |
|---------|--|-----------------|--|--|--|
| (1).... | sodium- $\alpha$ -naphthyl acetate.          | 0.001           | 423                                    | 74.8   | 28.3   |
| (2).... | sodium-2-methyl-4-chloro acetate.            | 0.001           | 414                                    | 71.1   | 24.9   |
| (3).... | 2,4-dichlorophenoxy acetic acid.             | 0.001           | 433                                    | 78.5   | 31.3   |
| (4).... | sodium phenyl acetate.                       | 0.005           | 430                                    | 77.7   | 28.5   |
| (5).... | indolyl acetic acid...                       | 0.001           | 433                                    | 78.9   | 29.1   |
| (6).... | control                                      | 0               | 245                                    | 43.8   | 21.4   |
| (7).... | control deficiently fed with animal protein. | 0               | 242                                    | 0  | 0  |

### Aspirin

An improved feed containing aspirin as a growth stimulant is reported by M.J. Caldwell (U.S. Patent 3,147,120; September 1, 1964; assigned to Moorman Manufacturing Company). When test animals are fed with a feed or ration containing small but effective concentrations of aspirin, while control animals are fed the same feed or ration without the aspirin, the former show greater increases in average daily gain in weight. Such increases have been noted particularly in the cases of ruminants and poultry.

Example: In the following table, the basic ration used had the composition consisting of mixed alfalfa-brome hay, free choice; shelled yellow corn, free choice; commercial cattle concentrate (commercially available protein-mineral concentrate containing 45% protein



## Growth-Promoting Chemical Additives

equivalent from oil meals and urea, 10% of a complex mineral mix, vitamins, 10 mg. of diethylstilbestrol per lb., and a small quantity of molasses), 1.0 lb./head/day.

**TABLE 1: ASPIRIN FOR FEEDING CATTLE \***

| Lot No. | Ration Fed   | Initial Weight, lb. | Final Weight, lb. | Average Daily Gain (140 Days), lb. | Weight Gain, lb. | Feed Efficiency (lb. feed/lb. gain), lb. |
|---------|--|---------------------|-------------------|------------------------------------|------------------|--|
| 1-----  | Basal Ration only.   | 610.9               | 972.6             | 2.58                               | 361.7            | 7.82                                     |
| 2-----  | Basal Ration plus 1 gram Aspirin (acetyl salicylic acid).  | 612.1               | 1,011.2           | 2.85                               | 399.1            | 7.46                                     |
| 3-----  | Basal Ration plus 5 grams Aspirin (acetyl salicylic acid). | 618.0               | 1,013.4           | 2.82                               | 395.4            | 7.64                                     |

\* Extra gains and feed efficiency from 1 or 5 g. aspirin per head per day.

### Chlorpromazine

It has been found by M.J. Lobel (U.S. Patent 3,171,745; March 2, 1965) that the addition of relatively minute amounts of chlorpromazine, when added to animal feeds, will accelerate the growth of grazing animals and domesticated fowl and their general well being, and will reduce the time for growth before they are marketable.

When used in large quantities in the amount that would be required to be effective as a tranquilizer, the accelerated growth is not observed and actually there is a contrary effect, as the poultry and grazing animals will lose weight rather than gain weight. The effective range is about 0.001 to 0.5 part by weight per 50,000 parts of feed with an optimum range of 0.003 to 0.25 part in weight. This is about 1/100 to 1/1000 of the normal dosage of chlorpromazine as would be used in connection with tranquilizing purposes.

However, chlorpromazine is not too effective in enhancing the growth factor when combined with feeds by itself. Chlorpromazine is most effective when combined in compositions including vitamin A and guaiacol, with the vitamin and guaiacol being used in about equal proportions and in the proportion of about 10 to 50 times the amount of chlorpromazine. In addition to the above ingredients, it is also possible to include camphor and iodine or sodium or potassium iodide in about the same amounts as guaiacol. The additional ingredients appear to enhance the growth factor.

The following is an example of a satisfactory composition:

| <u>Ingredient</u> | <u>Parts by Weight</u> | <u>Range</u>   |
|-------------------|------------------------|----------------|
| Vitamin A         | 1.112                  | 0.2224 to 2.78 |
| Iodine A          | 0.166                  | 0.033 to 0.5   |

(continued)

## Growth-Promoting Chemical Additives

(continued)

| <u>Ingredient</u> | <u>Parts by Weight</u> | <u>Range</u> |
|-------------------|------------------------|--------------|
| Camphor           | 1.33                   | 0.333 to 2   |
| Oil of eucalyptus | 1.33                   | 0.333 to 2   |
| Guaiacol          | 1.33                   | 0.333 to 2   |
| Chlorpromazine    | 0.17                   | 0.333 to 25  |
| Corn meal         | 480                    |              |

The amount of corn meal may be widely varied, for example, in feeds for poultry since it merely serves as a carrier, and it may be replaced by other inert or edible carriers such as charcoal, chalk, or even liquids such as glycerin or water in which the composition can be dissolved, dispersed, or emulsified.

The grazing animals and poultry which have been fed with the above additive have better bone structure, and their flesh is sweeter and better flavored. In the case of poultry comparative experiments show that where control poultry without the additive is compared in growth with poultry which is given the additive in a range of 0.05 to 2.0 ounces per hundred pounds of feed, the growth period preparatory to marketing may be shortened by 10%, and in many instances up to 20%.

### Rauwolfia Roots

H.G. Luther and J.R. De Zeeuw (U.S. Patent 3,092,496; June 4, 1963; assigned to Chas. Pfizer & Co., Inc.) found that a growth-promoting animal feed was obtained when Rauwolfia derived substances are added to a nutritionally balanced feed. The materials which are used to supplement the complete animal diets are the roots of certain species of the plant Rauwolfia, preferably in finely divided or ground form, in particular the species R. heterophylla, R. vomitoria, R. hirsuta, and so forth, which are generally sources of the alkaloid reserpine. Rather than utilizing the ground root of the plant, concentrates of the active material may be utilized for this purpose.

As little as 0.003% by weight of ground Rauwolfia root has a definitely beneficial effect upon the rate of growth of various nonruminant animals. In the case of ruminants, as little as 0.00005% of Rauwolfia root has a beneficial effect on growth. If a purified form of the Rauwolfia product is used to supplement the animal feeds, a proportionately smaller amount of the material may be used. In general, this is roughly in proportion of the reserpine content of the concentrate, although other materials contained in the Rauwolfia root apparently participate in the animal growth stimulation. Higher proportions of the ground root or its equivalent of more highly purified products may be utilized as a supplement in the animal feed. However, if a proportion appreciably higher than about 0.5% in the case of non-ruminants, and 0.05% in ruminants, calculated as ground root is used, there may be a lessening of the growth of the animals.

Example 1: For 14 weeks four groups of eight pigs each were fed a basal ration, and three of the four groups also had their diets supplemented with varying amounts of ground Rauwolfia vomitoria root. The accompanying table shows the results of this study.



## Growth-Promoting Chemical Additives

| <i>Rau. rom.</i> (percent by wt. feed) | Average<br>Daily<br>Gain (lbs.) | Growth<br>Index | Feed Effi-<br>ciency |
|--|---------------------------------|-----------------|----------------------|
| 0.....                                 | 1.63                            | 100             | 3.51                 |
| .0032.....                             | 1.73                            | 106             | 3.61                 |
| .032.....                              | 1.70                            | 104.5           | 3.52                 |
| .32.....                               | 1.68                            | 103.0           | 3.42                 |

The supplementation of animal diets with Rauwolfia root or equivalent products may conveniently be combined with supplementation by antibiotics and other growth stimulatory materials.

Example 2: One group of ten Hereford steers was fed a basal ration of supplement, together with 10 mg. per day per head of diethylstilbesterol and 80 mg. per day per head of oxytetracycline. A second group of ten comparable steers was fed the same ration, supplement, hormone and antibiotic, and in addition, 60 mcg. per day per head of reserpine. Results were as follows:

|                             | Control | Reserpine |
|-----------------------------|---------|-----------|
| <b>Growth:</b>              |         |           |
| Av. Initial Wt. (lbs.)..... | 751     | 746       |
| Av. Final Wt.....           | 1,068   | 1,106     |
| Av. Daily Gain.....         | 2.88    | 3.25      |
| Percent Increase.....       |         | 13        |
| <b>Feed Efficiency:</b>     |         |           |
| Lb. Feed/lb. gain.....      | 8.57    | 8.24      |
| Improvement, percent.....   |         | 4         |
| <b>Carcass Quality:</b>     |         |           |
| Dressing, percent.....      | 63.0    | 63.6      |
| Carcass Grade.....          | 9.7     | 11.6      |

### Straight Chain Aliphatic Alcohols

R.S. Gordon and L.J. Machlin (U.S. Patent 3,401,039; September 10, 1968; assigned to Monsanto Company) found that normal feed compositions for both ruminant and nonruminant animals can be made more effective if supplemented by the addition of linear aliphatic alcohols having from 4 to 22 carbon atoms. The alcohols may be either primary or secondary, but the primary alcohols are the most effective. Both saturated and unsaturated alcohols may be used, or mixtures as obtained by saponification of animal or vegetable oils followed by reduction of the carboxylic acids to the alcohols. Preferably the alcohol mixture so formed will contain at least 60% of the straight chain primary and secondary alcohols, not more than 20% of other alcohols, and not more than 20% by weight of nonalcoholic organic compounds.

0.01 to 12% by weight of the total feed will be the straight chain alcohols with 4 to 22 carbon atoms. Preferred practice may utilize from 1 to 8% by weight of these alcohols. To demonstrate the benefits to be derived from the process, feeding experiments were conducted.

Example: Well replicated lots of chicks at varying ages were fed a basal feed supplemented with the alcohols, and in some instances, with corn oil. The following table sets forth the observed data.

## Growth-Promoting Chemical Additives

| Parts added to 10 parts<br>of basal diet |          | Compound (name)     | Average<br>weight<br>gain (g.) |
|--|----------|---------------------|--------------------------------|
| Corn oil                                 | Compound |                     |                                |
| -----                                    | 0.0      | Control.....        | 91                             |
| -----                                    | 1.0      | n-Dodecanol.....    | 218                            |
| -----                                    | 1.0      | n-Octadecanol.....  | 115                            |
| -----                                    | 1.0      | n-Hexadecanol.....  | 158                            |
| -----                                    | 1.0      | n-Tetradecanol..... | 154                            |
| -----                                    | 1.0      | n-Docosanol.....    | 110                            |
| -----                                    | 1.0      | n-Pentanol.....     | 159                            |
| 2.0                                      | 0.5      | Isodecanol.....     | 146                            |
| 2.0                                      | 0.5      | 2-octanol.....      | 152                            |

Metabolism studies were conducted by feeding sheep conventional diets supplemented with 4% of the alcohols described in the following table. In these studies, measurements were made of percent of "fiber" in the feed which is assimilated by the animal, the percentage of "dry matter" which the animal digests, and the percent of the "nitrogen" of the feed assimilated by the sheep. The "nitrogen retention" is the grams per day of nitrogen retained by the animal, and is "indicative of the growth of protein tissue." The following table demonstrates the observed data on sheep of different age groups

| Alcohol         | Digestibility coefficients |            |          | Nitrogen<br>retained |
|-----------------|----------------------------|------------|----------|----------------------|
|                 | Fiber                      | Dry matter | Nitrogen |                      |
| Control.....    | 27.4                       | 63.3       | 80.5     | 7.00                 |
| Tetradecyl..... | 28.7                       | 64.0       | 83.5     | 12.10                |
| Decyl.....      | 26.2                       | 61.3       | 82.6     | 12.90                |
| Lauryl.....     | 35.2                       | 65.9       | 82.5     | 8.65                 |
| Control.....    | 30.7                       | 64.5       | 80.1     | 2.08                 |
| Oleyl.....      | 30.7                       | 62.6       | 79.9     | 5.56                 |
| Cetyl.....      | 30.7                       | 63.8       | 80.6     | 4.48                 |

### 2-Formyl-4-Chlorophenoxyacetic Acid

2-formyl-4-chlorophenoxyacetic acid and salts have growth promoting properties when added to animal feeds as described by J. Metivier and L. Julou (U.S. Patent 3,038,806; June 12, 1962; assigned to Societe des Usines Chimiques Rhone-Poulenc, France). The compositions are generally administered orally in an animal foodstuff, i.e., an organic or mineral substance which is intended to be fed to animals, or water. A dosage of 0.001% to 1% based on the total of foodstuffs and water given to the animal has been found to give satisfactory results, a dosage of 0.005 to 0.05% being preferred.

Example: Two groups of ten young male rates of homogeneous strain received for 4 weeks identical food which contained 50% of glucides, 25% of proteins, 3.5% of lipids, and 3% of mineral salts and vitamins, the remainder consisted of water. The treated rats received in addition 100 mg./kg. body-weight of 2-formyl-4-chlorophenoxyacetic acid, as an aqueous solution of the sodium salt, per day by oral administration. The concentration of the solution was such that the volume of solution administered was 5 cc/kg. The control rats received only pure water in the same proportion of 5 cc/kg.

The mean weight of the rats of each group was noted each week and the mean percentage weight increase was determined. The results are shown on the table on the following page. At the end of the experiment, the water content of the animals was determined. In the treated animals, a mean content of 62.1% was found, and in the controls, a mean content of 67.1%. This clearly showed not only that the gains in weight were not caused by



## Growth-Promoting Chemical Additives

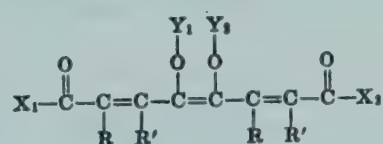
hydration of the tissue, but also that the active ingredient promoted the formation of new tissue.

| Group             | Mean weight at the start of the experiment, g. | Percent increase in the mean weight |         |         |         | Mean weight after 4 weeks, g. |
|-------------------|--|-------------------------------------|---------|---------|---------|-------------------------------|
|                   |  | 1 week                              | 2 weeks | 3 weeks | 4 weeks |                               |
| Treated rats..... | 150  | 15.3                                | 30.6    | 44.6    | 61.7    | 242.6                         |
| Control rats..... | 155  | 11.6                                | 15.4    | 17      | 37      | 212.4                         |

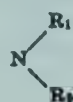
### 4,5-Dihydroxy-2,4,6-Octatrienedioic Acid Derivatives

The process of E.W. Bousquet and H.E. Hoffman (U.S. Patent 2,927,027; March 1, 1960; assigned to E.I. du Pont de Nemours and Company) relates to animal feed efficiency promoting compositions and methods employing as an essential active ingredient one or more compounds from the group including the salts, esters, amides, and cyclic monolactones and dilactones of 4,5-dihydroxy-2,4,6-octatrienedioic acid and its derivatives.

The active ingredients include compounds represented by the formula:



where R and R' are selected from the group consisting of hydrogen, alkoxyaryl, haloaryl, and monovalent hydrocarbon radicals free from nonaromatic unsaturation. X<sub>1</sub> and X<sub>2</sub> are selected from the group consisting of (1)



where R<sub>1</sub> and R<sub>2</sub> are selected from the group consisting of hydrogen, monovalent hydrocarbon radicals free from nonaromatic unsaturation, haloalkyl, aralkyl, alkoxy, aryl, haloaryl, and alkoxyaryl; (2) OR<sub>3</sub> where R<sub>3</sub> is selected from the group consisting of hydrogen, monovalent hydrocarbon radicals free from nonaromatic unsaturation, haloalkyl, aralkyl, alkoxy, aryl, haloaryl, and alkoxyaryl, provided that only one of the X<sub>1</sub> and X<sub>2</sub> radicals are OH in a single compound; and (3) OM where M is a cation. Y<sub>1</sub> and Y<sub>2</sub> are selected from the group consisting of hydrogen and alkali metal.

Also included are monocyclic and dicyclic derivatives where X<sub>1</sub> and Y<sub>1</sub>, or X<sub>2</sub> and Y<sub>2</sub> are removed to form the corresponding monolactone, or both of these combinations are removed to form the corresponding dilactone. Of the above compounds and cyclic derivatives, the di-gamma-lactone has particular advantages. The compounds and methods for their preparation are described in U.S. Patent 2,840,570.

Example 1: A standard pig primer diet consisting of 20% protein and made up mainly of corn, alfalfa meal, brown barley, soybean oil meal, supplemented with vitamins and yeast, was fed to two groups of male weanling pigs (Yorkshires), five animals per group. One

## Growth-Promoting Chemical Additives

group received basal diet only; the other group was fed the basal diet containing 0.1% by weight of 4,5-dihydroxy-cis-2,trans-4,cis-6-octatrienedioic acid, di-gamma-lactone. The experimental group receiving the feed efficiency promoter showed an improved feed efficiency at five and ten weeks. The data are as follows:

| Diet                            | Five Weeks                             |   | Ten Weeks                              |   |
|---------------------------------|--|---|--|---|
|                                 | Gain in Weight<br>(Average per animal) | Feed Efficiency<br>(Average per animal) | Gain in Weight<br>(Average per animal) | Feed Efficiency<br>(Average per animal) |
|                                 | <i>Kg.</i>                             |   | <i>Kg.</i>                             |   |
| Basal only.....                 | 39.85                                  | 2.32                                    | 96.00                                  | 2.87                                    |
| Basal with 0.1% di-lactone..... | 45.00                                  | 2.27                                    | 99.60                                  | 2.75                                    |

**Example 2:** Two groups each containing 32 one-day old male chicks of crossed strain (Lancaster males x Nichols #12 females) were fed a practical corn-soybean meal basal diet supplemented with vitamins and antibiotics. To one of the diets, 0.01% by weight of 4,5-dihydroxy-cis-2,trans-4,cis-6-octatrienedioic acid, di-gamma-lactone was added, while the other group received no such additive. The birds were maintained in wire-floored electrically heated brooders. At the end of four weeks, the chicks were weighed and feed consumption data recorded. The following figures were obtained:

| Diet                            | Gain in Weight<br>(Average per animal) | Feed Efficiency<br>(Average per animal) | Index of Performance<br>(Average gain of weight per animal/<br>Average feed efficiency per animal) |
|---------------------------------|--|---|--|
|                                 | <i>Grams</i>                           |   | <i>Grams</i>   |
| Basal only.....                 | 366                                    | 1.94                                    | 189  |
| Basal with 0.01% di-lactone.... | 372                                    | 1.86                                    | 200  |

**Example 3:** Two groups of Holstein calves, three males per group, are compared for eight weeks as to their feed consumption and weight gain. Both groups are fed calf starter with occasional supplements of leafy hay. To the diet of one group is added 0.01% by weight of 4,5-dihydroxy-cis-2,trans-4,cis-6-octatrienedioic acid, di-gamma-lactone. At the end of eight weeks, the calves receiving the growth stimulant according to this process show a gain of 7% more weight and demonstrate a marked improvement in feed efficiency on the basis of calf starter meal consumed per pound gained.

### 4,4'-Dihydroxydiphenylsulfide

The growth-promoting compositions of the process of H.M. Taylor (U.S. Patent 3,218,171; November 16, 1965; assigned to Eli Lilly and Company) are administered orally, and preferably comprise a major proportion of an ingestible carrier material and a minor, but physiologically adequate, proportion of 4,4'-dihydroxydiphenylsulfide.

**Example:** This experiment compares the effects of growth rate and feed efficiency of lambs receiving basal lamb ration (negative control) and lamb ration plus diethylstilbestrol (positive control) with lambs receiving lamb ration plus 4,4'-dihydroxydiphenylsulfide. Two milligrams



## Growth-Promoting Chemical Additives

per head per day of additive was used. The feeding procedure employed an experimental design of the block design type, utilizing a randomization method of allotment. The lambs employed were seven-month, Black-faced Texas wethers. (Each lamb was fed for a two-week conditioning period and then weighed, identified, and drenched with two ounces of phenoarsenate.) Each lot consisted of 10 lambs. Results of this experiment are summarized below:

|                                     | No. Animals | Days Fed | Av. Daily Gain, Lbs. | Feed Efficiency |
|-------------------------------------|-------------|----------|----------------------|-----------------|
| Negative Control <sup>1</sup> ..... | 20          | 47       | 0.48                 | 7.84            |
| Positive Control <sup>2</sup> ..... | 20          | 47       | 0.53                 | 6.74            |
| Test Material <sup>3</sup> .....    | 20          | 47       | 0.55                 | 6.82            |

<sup>1</sup> Basal ration.

<sup>2</sup> Basal ration plus diethylstilbestrol.

<sup>3</sup> Basal ration plus 4,4'-dihydroxydiphenylsulfide.

It can be seen that 4,4'-dihydroxydiphenylsulfide in feed at levels of 2 mg. per head per day improved rate of gain 13%, when compared to the negative control, basal ration (0.55 pound vs. 0.48 pound), and was closely equal to diethylstilbestrol (0.55 pound vs. 0.53 pound). Feed efficiency was improved 13%, when compared to the negative control (6.82 vs. 7.84 pounds of feed per pound of gain), and was also practically equal to diethylstilbestrol (6.82 vs. 6.74 pounds of feed per pound of gain). Likewise, in experiments on cattle, 4,4'-dihydroxydiphenylsulfide, when employed in concentrations of 5, 10 and 50 mg. per head per day, significantly improved rate of gain, when compared to the negative control.

Because of the nonhormonal and nonantibiotic character of 4,4'-dihydroxydiphenylsulfide, it is possible to administer this compound to animals over a wide range of dosage levels. Although no undesirable effects have been indicated at high dosage levels, it has been found that dosage levels ranging from about 2 to 20 grams per ton of the ingestible carrier material (e.g., animal feed or animal feed supplement) produce excellent results. For economic reasons, it is desirable to maintain dosage levels as low as is compatible with maximum growth stimulation. Therefore, it is preferred to employ dosage levels of no greater than 10 grams per ton of ingestible carrier. 4,4'-dihydroxydiphenylsulfide can conveniently be administered to the growing animal in dosages based on body weight. It is preferred to employ a dosage ranging from about 0.1 to 5 mg. of 4,4'-dihydroxydiphenylsulfide per 100 pounds of body weight over each 24-hour period.

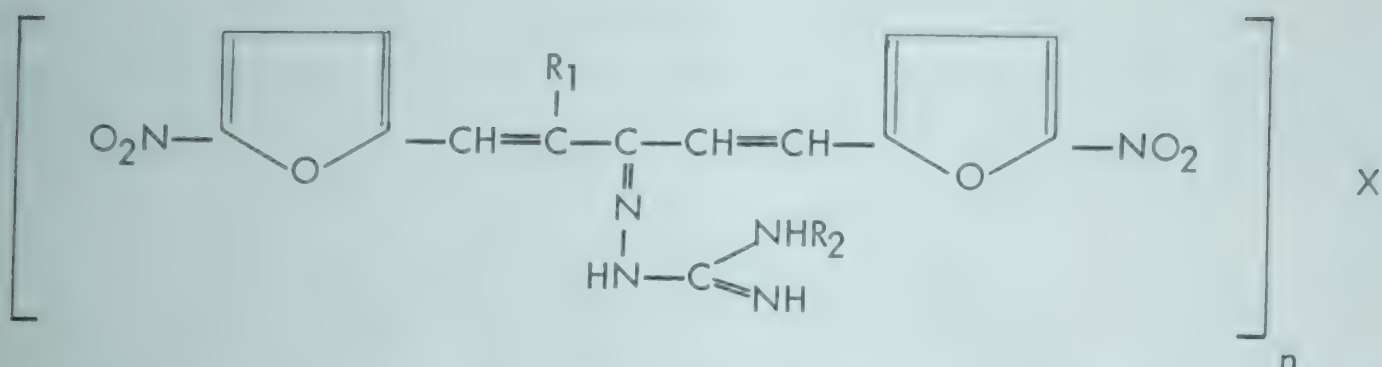
On the basis of feed intake, when 4,4'-dihydroxydiphenylsulfide is incorporated in the total feed ration (that is, in an animal feed nutritionally adequate per se), the feed material generally can contain from about 0.01 to 8 mg. per pound of feed material. However, it is preferred to employ from about 0.1 to 5 mg. per pound of feed material.

### Bis-(5-Nitrofurfurylidene)-Acetoneguanylhyazone

The process of Y. Kodama, T. Fujiwara, and T. Inagaki (U.S. Patent 3,264,112; August 2, 1966; assigned to Toyama Kagaku Kogyo K.K., Japan) is concerned with a growth-promoting additive used with conventional nutritionally balanced diets.

The additive is a bis-(5-nitrofurfurylidene)-acetoneguanylhyazone and its addition salts such as the hydrochloride. The formula is as shown on the following page.

## Growth-Promoting Chemical Additives



In the above formula  $\text{R}_1$  is hydrogen, lower alkyl or halogen;  $\text{R}_2$  is hydrogen or lower alkyl;  $\text{X}$  is a mono-, di-, or tribasic acid, either inorganic or organic;  $n$  is 1, 2, or 3 with the proviso that when  $\text{X}$  is monobasic  $n$  is 1, when  $\text{X}$  is dibasic  $n$  is 1 or 2, and when  $\text{X}$  is tribasic  $n$  is 1, 2 or 3. The unsubstituted derivative in which  $\text{R}_1$  and  $\text{R}_2$  are hydrogen and  $\text{X}$  is chlorine is sold in the trade under the name Panazon and for simplicity the short term will be used, it being understood that it is used only in the above meaning.

The additives to the feeds may be used over a broad range, for example, from about 5 to 500 ppm. Optimum results are obtained, particularly with chicks, at a concentration nearer 25 ppm.

**Example 1:** In each group Peterson-cross broiler chicks were used, six males and six females per group. Feed and water were supplied ad libitum. The effect of varying amounts of Panazon and procaine penicillin was tested; the diet, a typical commercial chick feed, was used as a control; and the drugs were added in separate tests in amounts of 12.5, 25, 50, and 100 ppm. The following table shows the results.

| Compound                  | Age           | Growth Response Percent of Control |      |     |     |     |
|---------------------------|---------------|------------------------------------|------|-----|-----|-----|
|                           |               | p.p.m.                             |      |     |     |     |
|                           |               | 0                                  | 12.5 | 25  | 50  | 100 |
| Panazon.....              | 1 week .....  | 100                                | 116  | 127 | 111 | 120 |
|                           | 2 weeks ..... | 100                                | 105  | 116 | 106 | 107 |
|                           | 3 weeks ..... | 100                                | 105  | 112 | 102 | 104 |
|                           | 4 weeks ..... | 100                                | 105  | 110 | 103 | 103 |
| Procaine Penicillin ..... | 1 week .....  | 100                                | 111  | 119 | 107 | 131 |
|                           | 2 weeks ..... | 100                                | 100  | 107 | 103 | 116 |
|                           | 3 weeks ..... | 100                                | 97   | 103 | 102 | 105 |
|                           | 4 weeks ..... | 100                                | 98   | 101 | 103 | 104 |

It will be seen that the optimum amount of Panazon is about 25 ppm and at this concentration there was markedly better results than with any concentrations of the antibiotics after four weeks. Even when 100 ppm of Panazon is compared with penicillin at the same figure, their optimum, the results were not significantly different at the end of four weeks. In other words, Panazon is as effective as penicillin and in its preferred concentrations, which is lower, is more effective.

**Example 2:** The effectiveness of feeds containing Panazon in preventing infection was tested with four groups of 80 male Leghorn chicks. The standard diet was used and the feeding period was extended for nine weeks under conditions which did not involve any attempt to protect the chicks from coccidiosis infection. The large numbers in each group



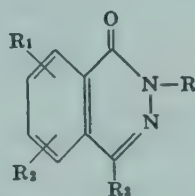
## Growth-Promoting Chemical Additives

were used because the incidence of coccidiosis is not so high that one could be sure that there would be cases in a smaller group. The results appear on the following table. It will be seen that beginning at the fifth week the controls showed occurrence of coccidiosis, but the chicks which were fed the feeds with varying amounts of Panazon showed no cases. It is apparent, therefore, that substantial protection against infection is obtained.

| Group     | Number of symptomatic chicks | Period after treatment started (weeks) |   |   |   | Total | Percent of symptomatic chicks |
|-----------|------------------------------|--|---|---|---|-------|-------------------------------|
|           |                              | 3                                      | 5 | 7 | 9 |       |                               |
| Control   |                              | 0                                      | 1 | 2 | 0 | 3     | 3.75                          |
| 10 p.p.m. | (80)                         | 0                                      | 0 | 0 | 0 | 0     | 0                             |
| 20 p.p.m. | (80)                         | 0                                      | 0 | 0 | 0 | 0     | 0                             |
| 40 p.p.m. |                              | 0                                      | 0 | 0 | 0 | 0     | 0                             |

### Phthalazinone Derivatives

A. Margot (U.S. Patent 3,433,641; March 18, 1969; assigned to Geigy Chemical Corp.) found that phthalazinone derivatives are useful as growth-promoting additives. These are of the formula:



in which R is hydrogen, lower alkyl, the group:  $-\text{C}(=\text{O})\text{R}_4$  (R<sub>4</sub> being alkoxy or alkenyloxy),

or the group:  $\text{alkylene}-\text{C}(=\text{O})\text{R}_5$  (alkylene having 1 to 3 carbon atoms and R<sub>5</sub> being amino, lower alkyl amino, di-lower alkyl amino, or the group OX where X is hydrogen or the nontoxic cation of a base);

R<sub>1</sub> and R<sub>2</sub> independently are hydrogen, halogen, nitro or lower alkyl; and R<sub>3</sub> is lower alkyl, halogen or the group OY where Y is hydrogen or the nontoxic cation of a base. They can be produced by known methods.

Feed additive concentrates are produced which contain 1 to 50%, and preferably 5 to 10% of active substance, whereas the finished feed advantageously contains the active substances in a concentration of, e.g., 0.01 to 0.03%.

**Example 1:** 50 parts of 2-methyl-5-chloro-4-hydroxy-1(2H)-phthalazinone, 0.8 part of sodium benzoate, 2 parts of tylose, 5 parts of sorbitan monooleate and 42.2 parts of white flour are intimately mixed to form a feed additive. A 50% feed concentrate is obtained which can be mixed to any concentration desired with water, milk, etc.

**Example 2:** A group of 15 pigs were fed, per animal and per day, with 7 liters of buttermilk (5 to 6% dry substance) and 1 to 2 kg. of a dry mixed feed of the following composition.

## Growth-Promoting Chemical Additives

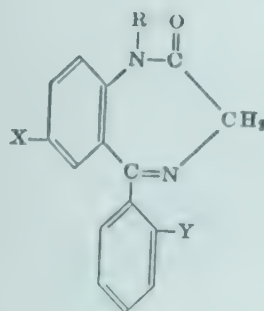
| <u>Ingredient</u>               | <u>Kg.</u> |
|---------------------------------|------------|
| Barley                          | 56         |
| Maize                           | 9          |
| Milo                            | 9          |
| Wheat/rye                       | 8          |
| Bran                            | 10         |
| Hay meal                        | 4          |
| Dried potato                    | 10         |
| Mineral substances and vitamins | 2          |

To the above composition was added, per animal and per day, 100 mg. of 2-methyl-4-hydroxy-1(2H)-phthalazinone. The increase in weight was compared with that of an equal sized control group fed with the same feed but without active substance. The results are shown below.

|                                   | With active substance added (kg.) | Control group (kg.) |
|-----------------------------------|-----------------------------------|---------------------|
| Average initial weight.....       | 39.8                              | 41.2                |
| Average weight after 30 days..... | 54.3                              | 51.0                |
| Increase in weight.....           | 14.5                              | 9.8                 |

### Benzodiazepinone Derivatives

The growth-promoting agents employed in the feed compositions and supplements of the process of J.C. Bauernfeind (U.S. Patent 3,248,223; April 26, 1966; assigned to Hoffmann-La Roche Inc.) have the following formula:



where X is nitro or halogen; R is hydrogen or lower alkyl, preferably methyl; and Y is hydrogen, trifluoromethyl, or halogen. The term "halogen" used above for X and/or Y includes fluorine, chlorine, bromine, or iodine, with chlorine preferred.

Examples of compounds having the above structure include:

- 7-chloro-5-phenyl-1-methyl-3H-1,4-benzodiazepin-2(1H)-one
- 7-nitro-5-( $\alpha,\alpha,\alpha$ -trifluoro-o-tolyl)-3H-1,4-benzodiazepin-2(1H)-one
- 7-nitro-5-(2-chlorophenyl)-3H-1,4-benzodiazepin-2(1H)-one

and so forth. They are employed in the feed supplement or finished feed in an amount that will supply the animal with a daily intake of about 1 to 100 mg., preferably 2 to 50 mg., per head per day.



## Growth-Promoting Chemical Additives

Example 1: 600 lb. feeder cattle were fed with the feed ration shown for a 120 day feeding period, and were allowed to consume as much feed as they desired. A control high grain-part roughage ration consisted of the following ingredients:

| <u>Ingredients</u>         | <u>Percent</u> |
|----------------------------|----------------|
| Barley                     | 60             |
| Molasses                   | 10             |
| Soybean-mineral supplement | 10             |
| Alfalfa hay                | 20             |
|                            | <u>100</u>     |

The results are shown below.

| <u>Groups—10 cattle per group</u>   | <u>Gain in wt. (lb./head/day)</u> | <u>Feed utilization (lb. feed/lb. gain)</u> |
|---|-----------------------------------|---|
| Control ration .....  | 2.80                              | 9.0   |
| Control ration plus 10 mg. 7-chloro-5-phenyl-1-methyl-3H-1,4-benzodiazepin-2(1H)-one <sup>1</sup> per head per day .....                      | 3.15                              | 8.6   |
| Control ration plus 10 mg. 7-nitro-5-( $\alpha,\alpha$ -trifluoro-o-tolyl)-3H-1,4-benzodiazepin-2(1H)-one <sup>1</sup> per head per day ..... | 3.10                              | 8.5   |
| Control ration plus 10 mg. 7-nitro-5-(2-chlorophenyl)-3H-1,4-benzodiazepin-2(1H)-one <sup>1</sup> per head per day .....                      | 3.10                              | 8.6   |

<sup>1</sup> Fed mixed in the soybean-mineral supplement.

The growth-promoting agents of this process can also be used in conjunction with other feed additives, e.g., antibiotics, hormones, enzymes, arsenicals, coccidiostats, etc.

Example 2: Young turkey poults were fed the following control growing ration for a study period of 16 weeks:

| <u>Ingredients</u>          | <u>Percent</u> |
|-----------------------------|----------------|
| Corn meal                   | 38             |
| Soybean oil meal            | 37             |
| Middlings                   | 5              |
| Fish meal                   | 5              |
| Meat scraps                 | 5              |
| Fat                         | 2              |
| Alfalfa                     | 2              |
| Distillers solubles         | 3              |
| Mineral vitamin supplements | 3              |
|                             | <u>100</u>     |

|                        |                  |
|------------------------|------------------|
| Furazolidone (NF-180)  |                  |
| (in supplement)        | 0.010% of ration |
| Amprol (in supplement) | 0.010% of ration |

## Growth-Promoting Chemical Additives

The results of this study are shown on the following table.

| Groups  | Gain in wt. (wt. in lbs.) | Feed utilization (lb. feed/lb. gain) |
|---|---------------------------|--------------------------------------|
| Control nitrofurantoin-coccidiostat ration.....   | 14.5                      | 3.0                                  |
| Control nitrofurantoin-coccidiostat ration plus 25 mg. 7-chloro-5-phenyl-1-methyl-3H-1,4-benzodiazepin-2(1H)-one per lb. of ration..... | 16.0                      | 2.8                                  |

### Maltol (3-Hydroxy-2-Methyl- $\alpha$ -Pyrone)

It has been found by W.A. Olson (U.S. Patent 3,338,718; August 29, 1967; assigned to Chas. Pfizer & Co., Inc.) that when maltol, which is chemically known as 3-hydroxy-2-methyl- $\alpha$ -pyrone, is incorporated into feeds, there is obtained a substantially greater growth increase response in animals than when the feeds are used alone. For these purposes a concentration level as low as 5 grams per ton of maltol with respect to the animal feed is generally sufficient to impart a substantial growth response to the animal. In general, maltol can be employed in the feeds at concentration levels ranging from about 5 to 200 grams per ton of feed in order to achieve the unusually high degree of growth promotion previously referred to.

The preferred proportion is in the concentration range of from about 20 to 100 grams per ton of feed, although this will vary to some extent depending upon the animal's weight, its individual response to the growth promoter, and the particular species of animal that is being treated. It has usually been found convenient in practice to administer the maltol in the form of the animal's feed, i.e., on mixture with the feed.

Example 1: Forty-eight 20 to 24 day old baby pigs were divided into six pens of eight animals each (four male and four female) and fed a standard nutritious diet suitable for early weaned pigs containing approximately 20% crude protein, as well as oxytetracycline at the 200 g. per ton concentration level, and also including cane sugar (sucrose) at a level of 7.5% by weight. One group of 16 such pigs was fed this ration alone and served as the control, while the other two such groups each received this ration together with maltol present in the feed at concentration levels of 25 g. per ton and 200 g. per ton, respectively.

All the pigs were individually weighed initially as well as at the end of three and five-week periods of feeding (post-weaning). The results obtained in this manner, with respect to animal growth are summarized below, where the growth index values (percent increases) are given in parentheses in the table. As indicated by the data presented, the growth of pigs was favorably influenced by the addition of maltol to the feeds at the two different concentration levels tested.

| Treatment            | Average Weight Gain, lbs./pig |             |
|----------------------|-------------------------------|-------------|
|                      | 6 weeks                       | 8 weeks     |
| Control.....         | 8.2(100)                      | 21.6(100)   |
| Maltol, 25 g./T..... | 10.6(129.1)                   | 28.1(116.2) |



### Piperazine Phosphate

It is known that piperazine and its salts and derivatives are effective anthelmintic agents when ingested at relatively high dosages. P.C. Hereld (U.S. Patent 2,990,283; June 27, 1961; assigned to Polychemical Laboratories, Inc.) found that the combination of piperazine phosphate with the usual feed of animals at subascaricidal and subanthelmintic levels has surprising and unexpected results in increasing the growth rate and weight gain of the animals and in bringing out more efficient conversion of the feed into weight gain.

Piperazine phosphate is mixed and combined with any of the usual animal feeds in such amount that it is present in the feed to the extent of 200 to 1,500 grams per ton. The lower limit of 200 grams per ton has been found to be of critical significance because, while some minor deviation from this amount is permissible, amounts materially less than 200 grams per ton have been determined to be inadequate. The upper limit is well below the minimum amount used for ascaricidal and anthelmintic action. Approximately 400 grams (less than 1 pound) per ton has been found to be best when all factors are considered, and is thus preferred. This represents less than one-fourth of the minimum effective ascaricidal and anthelmintic dosage of 4 pounds per ton.

Example: Tests were carried out on weanling pigs fed with a basal ration as a control group and with the same basal ration with which piperazine phosphate was combined in the proportion of 20 grams of piperazine phosphate per 100 pounds of feed. The basal ration consisted depending upon the body weight of the pigs, of 77 to 84% ground yellow corn, 20 to 13% soybean oil meal, and minor percentages (0.05 to 1.0%) of ground limestone, steamed bone meal, iodized salt, trace minerals, vitamin supplement (2-49 C Lederle) and antibiotic supplement (TM-10 Pfizer), all percentages being by weight.

The control weanling pigs fed the basal ration showed an average daily weight gain, from the average initial weight of 25.9 pounds to the average final market weight of 185.7 pounds, of 1.57 pounds whereas the pigs fed the basal ration plus 20 grams of piperazine phosphate per 100 pounds of feed showed an average daily weight gain of 1.69 pounds and a final average market weight of 197.5 pounds, even though the average initial weight was 25.6 pounds. The control group of pigs consumed on the average 4.71 pounds of feed daily whereas the pigs fed the basal ration plus the piperazine phosphate (20 g./100 lbs. of feed) consumed on the average 5.20 pounds of feed daily. The tests were conducted during a period of 102 days.

### 1-(p-Chlorobenzhydryl)-4-Methylpiperazine Hydrochloride

It has been found that the growth of animals can be accelerated by providing in the various commercial animal feed compositions certain amounts of 1-(p-chlorobenzhydryl)-4-methylpiperazine hydrochloride, sold under the trademark Di-Paralene. J.L. Schmidt, H.S. Perdue, G.F. Lambert and J.P. Miller (U.S. Patent 2,997,393; August 22, 1961; assigned to Abbott Laboratories) found that the addition of this compound appears to stimulate the appetite of the animal and thereby causes an increase in weight and growth. This effect is observed by the fact that animals given a diet to which Di-Paralene is added, eat more of the diet than do those given a normal basal diet.

Example: Thirty 1-day old cockerels of the White Rock variety were fed a basal diet that

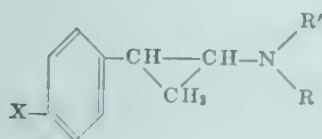
## Growth-Promoting Chemical Additives

contained 10 g./ton of Di-Paralene and at the end of a 4-week feeding period, the average weight of the chicks was 495.1 g./chick. Thirty other chicks of the same variety and age were fed a basal diet that contained 100 g./ton of Di-Paralene and at the end of a 4-week feeding period, their average weight was 508.1 g./chick. The two groups fed Di-Paralene in this amount showed a marked increase in weight over a control group of thirty other chicks of the same variety and age for a 4-week feeding period, which had an average weight of only 475.7 g./chick.

This process includes the administration of Di-Paralene in forms other than as a solid in feed mixes; thus, for example, the growth of animals can be promoted by adding Di-Paralene to their drinking water. Growth is best promoted when 1 to 10 grams of Di-Paralene are added for every 50 gallons of water. Satisfactory premixes of Di-Paralene may be made with a variety of naturally occurring materials and with any desired concentration of Di-Paralene. Examples of suitable premixing agents are soybean oil meal, ground limestone, oyster shell flour, corn meal, soybean mill feed, ground milo maize and other grains and feed ingredients.

### 2-Phenylcyclopropylamine

The compositions used as growth additives in the process of T. Ellison (U.S. Patent 3,134,676; May 26, 1964; assigned to Smith Kline & French Laboratories) are characterized in that they contain small amounts of a compound or compounds having the formula:



where X may be hydrogen, halogen, or trifluoromethyl; R and R' may be hydrogen or lower alkyl.

Desired results are realized when these compounds are administered in concentrations of from 1 to 100 parts per million, and preferably in the range from 5 to 50 ppm. At these extremely low levels, a surprisingly high increase in rate of growth is observed. The compositions may be prepared by mixing the compound directly with the basal feed ration itself or, alternatively, by employing a suitable premix composition which is introduced to and mixed in known quantities with a standard basal feed ration.

Example: The following ingredients are thoroughly mixed and employed as a feed composition for chickens.

| <u>Ingredients</u>           | <u>Quantity</u> |
|------------------------------|-----------------|
| Ground yellow corn           | 1,139 lbs.      |
| Stabilized fat               | 120 lbs.        |
| Fish meal, 60%               | 125 lbs.        |
| Soybean meal, 50%            | 480 lbs.        |
| Corn gluten meal             | 50 lbs.         |
| Dehydrated alfalfa meal, 20% | 25 lbs.         |
| Limestone                    | 20 lbs.         |

(continued)



## Growth-Promoting Chemical Additives

(continued)

| <u>Ingredients</u>                              | <u>Quantity</u> |
|---|-----------------|
| Dicalcium phosphate                             | 28 lbs.         |
| Salt  | 6 lbs.          |
| Trace mineral mix <sup>1</sup>                  | 2 lbs.          |
| Vitamin-amino acid mix <sup>2</sup>             | 5 lbs.          |
| Trans-2-phenylcyclopropylamine<br>hydrochloride | 18.18 grams     |

<sup>1</sup> Trace mineral mix contains manganese 60 p.p.m.; iodine 1.2 p.p.m.; copper 2 p.p.m.; cobalt 4 p.p.m.; iron 25 p.p.m. and zinc 18 p.p.m.

<sup>2</sup> Vitamin-amino acid mix contains riboflavin 4 g.; niacin 30 g.; calcium pantothenate 10 g.; choline chloride 1.5 lbs.; vitamin B<sub>12</sub> (1000 mcg./g.) 6 g.; vitamin A acetate (500,000 U.S.P. units/g.) 6 g.; vitamin D<sub>2</sub> (850,000 I.C.U./g.) 1 g.; vitamin E acetate (250 I.U./g.) 5 g.; DL-methionine 1.0 lb.; corn meal to a total of 5 lbs.

A growth study was conducted using the feed composition described above as the feed for fifty Vantress X Arbor Acre cockerels. An identical basal feed ration without 2-phenylcyclopropylamine was employed as a control and administered to an additional fifty cockerels. The following table shows the results of this study.

| Weeks  | Control               |          | 2-Phenylcyclopropylamine Hydrochloride |          | Increase over Control, percent |
|--------|-----------------------|----------|--|----------|--------------------------------|
|        | Average Total Wt., g. | Gain, g. | Average Total Wt., g.                  | Gain, g. |                                |
| 0..... | 76.8                  | -----    | 76.7                                   | -----    | -----                          |
| 1..... | 180.6                 | 103.8    | 176.7                                  | 100.0    | -----                          |
| 2..... | 310.5                 | 233.7    | 325.4                                  | 248.7    | 6.4                            |
| 3..... | 425.2                 | 348.4    | 469.8                                  | 393.1    | 12.8                           |
| 4..... | 472.9                 | 396.1    | 574.7                                  | 498.0    | 25.7                           |

### Rennet Plus Calcium Chloride

A growth-promoting additive to balanced diets was found in rennet activated with calcium chloride by S. Schöner (U.S. Patent 3,222,179; December 7, 1965; assigned to Fuv AG, Liechtenstein). A mixture of rennet and calcium chloride at a proportion of 1 to 5, amounts of 100 to 150 grams per 100 kg. of feed proved to be very successful. The following examples illustrate the use of this additive.

Example 1 — Hogs: A conventional feeding consisting of a normal fodder of a good quality with a total content in nutritive substances of, for example, 700 to 750 g./kg., and without the additive, was provided. With this feeding, the normal feeding period for producing an increase of 100 kg. in live weight amounts to 130 to 180 days. The total amount of fodder required for an increase of 100 kg. in live weight then lies between 300 kg. in the most favorable case and 450 kg. in the most unfavorable case.

When the additive is included in the normal fodder, according to the tests which were carried out, the normal feeding period amounted to 110 to 150 days. The total amount of fodder required for an increase of 100 kg. in live weight lies between about 160 kg. in the most favorable case, and 260 kg. in the most unfavorable case. A comparison of the

## Growth-Promoting Chemical Additives

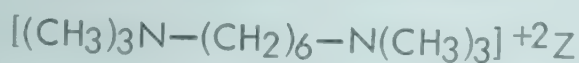
results shows that when applying the feed additive a saving of about 40% and more in fodder will be attained. Furthermore, it has been found that with a fodder containing the additive, the animals are very quickly satisfied which means that considerably smaller amounts of the fodder are required than by the usual fodder. The tests in which the above results were found also showed that the meat component was increased and the fat component was reduced.

Example 2 — Poultry: For producing an increase of 1 kg. in the live weight of a chicken, 2.2 to 2.8 kg. of a ready-made, industrially prepared chicken feed were used. Similar feed mixtures with the additive (rennet to calcium chloride at a ratio of 1 to 5, and with 150 g. of this mixture per 100 kg. of feed) resulted in the tests in a consumption of only 1.25 kg. of feed, and in some cases in one of only 1 kg. of feed per each kg. of increased live weight. The force-feeding period with poultry feed amounted to 56 days which resulted in a live weight of each animal of 1,000 to 1,250 g. The same results were attained in the tests in only 45 days when the feed additive was applied. This means a saving in feed of 50% and more and a reduction of the force-feeding period of about 20%.

Example 3 — Milk Fed Calves: For producing an increase of 1 kg. in live weight, 13 kg. of skimmed milk are generally required. When applying rennet alone as an addition to the milk, the amount of milk could be reduced from 13 kg. to 11 kg. When the new feed additive containing calcium chloride or calcium acetate and the same amount of rennet was added to the milk, the amount of milk required could be reduced from 13 kg. to 8 kg. With rennet alone, only 15%, and with the new feed additive about 40% of milk was saved.

### Hexamethonium Salts

Compositions for increasing the growth and feed efficiency of quadruped animals, both ruminant and nonruminant, comprising nutritionally balanced animal feeds containing minor proportions of nontoxic hexamethonium salts of the formula:



where Z is a nontoxic anion and is present in sufficient molar proportion to satisfy electro-neutrality, are described in the process of F.A. Hochstein (U.S. Patent 3,397,990; August 20, 1968; assigned to Chas. Pfizer & Co., Inc.).

In administering the products it is preferred to incorporate these materials in a balanced animal diet. However, equally beneficial effects in growth rate and feed efficiency are obtained by simply adding the hexamethonium salt to the animals' drinking water or, where appropriate, to salt licks, or by direct oral administration. In general it is preferred to use the hexamethonium salts at a concentration of at least 20 mg. of the salt per kg. of the total feed supplied the animal. In some cases, a somewhat higher level is useful with a particular species of animal or when a particular salt is used. A range of from about 20 mg./kg. to 500 mg./kg. of feed, corresponding to about 0.002 to 0.05% (w./w.), or about 18 g. to 500 g./ton of feed, seems to be effective in producing the beneficial effects.

Example: Three groups of weanling Sprague-Dawley rats, each group containing 5 male and 5 female rats, and a control group containing 10 rats of each sex are used in this experiment. The rats are individually housed in wire-bottom cages. Water and food are provided



## Growth-Promoting Chemical Additives

ad libitum throughout the 8-week experimental period. The basal ration containing 0.1% Delamix (trademark name for a trace mineral premix) was used. The central group is fed this basal ration. The three treatment groups are fed the basal ration to which hexamethonium chloride (HMT-Cl) is added at the expense of the corn meal in the ration in varying concentrations. The rats are weighed initially and then every two weeks throughout the eight weeks of the experiment. Feed intakes are determined every other day.

The concentrations of hexamethonium chloride and the results obtained are tabulated below. The "Gains Index" is defined as:

$$\frac{\text{Average gain produced by the supplemented feed}}{\text{Average gain produced by the unsupplemented feed}} \times 100$$

The unsupplemented feed or control group is arbitrarily assigned a Gains Index of 100.

| Supplement    | Avg. Gain gms./rat | Gains Index |
|---------------|--------------------|-------------|
| None          | 43.8               | 100         |
| HMT-Cl 0.002% | 66.6               | 152         |
| HMT-Cl 0.01%  | 58.2               | 133         |
| HMT-Cl 0.05%  | 64.1               | 146         |

It is obvious from this data that the addition of as little as 0.002% of hexamethonium chloride added to the animal's diet induces an appreciable increase in the rate at which weight is gained.

### Gamma Amino Butyric Acid as Appetite Depressant

For certain purposes it is desired to restrict the voluntary feed intake of animals to achieve particular results, yet it is desired to do this without harm to the animals and while maintaining adequate nutrition. In a process by R. Teichman and J.P. Everett, Jr. (U.S. Patent 3,408,200; October 29, 1968; assigned to Ralston Purina Company), animal feeds are provided which afford adequate nutrition for animals, which may be effectively utilized by the animals for growth and production purposes, and yet which decrease the rate of voluntary feed intake.

Although otherwise conventional in components, the feeds of this process contain as an essential ingredient a comparatively small proportion of gamma amino butyric acid, as small as 0.68%. Increase in the proportion of gamma amino butyric acid above 0.68% by weight of the other feed components will result in a substantially linear reduction of voluntary feed consumption. The following examples illustrate the process.

Example 1: Two groups of 32-week old broiler type chicks were fed under controlled conditions with a standard broiler starting feed for three weeks and then with a standard finisher feed for the following five weeks. One group functioned as the control group and was fed these feeds in the customary manner. The other group was offered the same feeds but into which was incorporated 0.68% by weight of gamma amino butyric acid.

The results secured are shown on the table on the following page.

## Growth-Promoting Chemical Additives

|   | Control, g. | Gamma Amino Butyric Acid, g. |
|---|-------------|------------------------------|
| Initial Liveweight.....                                 | 256         | 245                          |
| Final Liveweight <sup>1</sup> .....                     | 1,527       | 1,453                        |
| Liveweight Gain.....                                    | 1,278       | 1,176                        |
| Feed Consumption <sup>1</sup> .....                     | 2,822       | 2,785                        |
| Gamma Amino Butyric Acid Consumption <sup>1</sup> ..... | 0           | 18.55                        |

<sup>1</sup> Adjusted by covariance for Initial Liveweight.

**Example 2:** Example 1 was repeated but the feeds contained 2.04% by weight of gamma amino butyric acid. The results secured are shown below.

|   | Control, g. | Gamma Amino Butyric Acid, g. |
|---|-------------|------------------------------|
| Initial Liveweight.....                                 | 256         | 248                          |
| Final Liveweight <sup>1</sup> .....                     | 1,527       | 1,362                        |
| Liveweight Gain.....                                    | 1,278       | 1,094                        |
| Feed Consumption <sup>1</sup> .....                     | 2,822       | 2,634                        |
| Gamma Amino Butyric Acid Consumption <sup>1</sup> ..... | 0           | 53.00                        |

<sup>1</sup> Adjusted by covariance for Initial Liveweight.

Replacement of these feeds with conventional animal feeds will result in subsequent voluntary feed intake at a substantially normal rate.

**Example 3:** The test groups of Example 1, after the test period, were both fed for three additional weeks with the standard broiler-finisher feed which did not contain any gamma amino butyric acid. The results secured are shown below.

|                                     | Control, g. | Test Group, g. |
|-------------------------------------|-------------|----------------|
| Initial Liveweight.....             | 1,527       | 1,453          |
| Final Liveweight <sup>1</sup> ..... | 2,287       | 2,266          |
| Liveweight Gain <sup>1</sup> .....  | 882         | 861            |
| Feed Consumption <sup>1</sup> ..... | 2,307       | 2,461          |

<sup>1</sup> Adjusted by covariance for Initial Liveweight.

## POULTRY AND SWINE

### Sodium N-Glycolylarsanilate

The process of E.W. Dennis and A. Arnold (U.S. Patent 3,271,159; September 6, 1966; assigned to Sterling Drug Inc.) provides an arsenic-containing feed composition for promoting the growth and improving the outward appearance of poultry and swine safely, and, in particular, without leaving toxic levels of trivalent inorganic arsenic in the tissues of the animals. This can be accomplished by orally administering to the poultry and swine novel feed compositions containing an effective growth-promoting and appearance-improving quantity of sodium N-glycolylarsanilate.

A feed composition which provides optimum effects of improved rate of growth, improved feed conversion and improved appearance in poultry can best be obtained from a feed composition comprising from about 20 to 40 grams of sodium N-glycolylarsanilate per ton of feed (about 0.002 to 0.004% by weight). Administration of the medicated feeds is preferably begun immediately after the poultry are hatched and maintained during the entire



## Growth-Promoting Chemical Additives

growing period of the birds. Swine feeds which contain from about 15 to 45 grams of sodium N-glycolylarsanilate per ton of feed (about 0.0015 to 0.0045%) are useful for promoting the growth and for improving the feed conversion of young pigs.

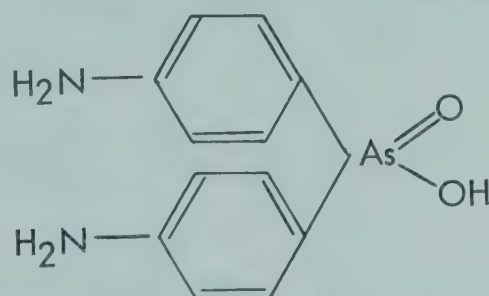
Example: Four lots of white Vantress X Nichols 108 straight run chicks containing 120 chicks per lot were fed basic poultry feed containing a commercial coccidiostat consisting of 50% bithionol [2,2'-thiobis(4,6-dichlorophenol)], 10% methiotriazamine [4,6-diamino-1-1(4-methylmercaptophenyl)-1,2-dihydro-2,2-dimethyl-1,3,5-triazine hydrochloride], and 40% calcium sulfate. Sodium N-glycolylarsanilate in the amount of 30 grams per ton of feed was added to the diet of two of the four lots and the remaining two lots were maintained as controls. After eight weeks, the average weight of the medicated birds was 1,393 grams, while the average weight of the untreated birds was 1,304 grams. The feed conversion of the treated birds was 1.86, and of the untreated birds, 1.70.

The chickens receiving the arsenic-medicated feed had an improved appearance over the control chickens. Specifically, the treated chickens showed an improved sheen and smoothness of the feathers, a deepening of the red color of the comb and wattles, and a deepening of the pigmentation of the shank and of the skin of the legs.

The tissues of the treated chickens contained no arsenic compounds in the objectionable trivalent form. Small quantities of arsenicals were found in the tissues, and in particular, in the liver, but the arsenic therein was exclusively in the relatively nontoxic pentavalent form. The tissues of chickens given a five-day withdrawal period contained virtually no arsenical residues; however, in view of the absence of detectable inorganic residual arsenic, even when no withdrawal period was used, such withdrawal is unnecessary.

### Diamino Diphenyl Arsinic Acid

J.R. Wiley and T.S. Chang (U.S. Patent 3,264,111, August 2, 1966; assigned to Whitmoyer Laboratories, Inc.) found that diamino diphenyl arsinic acid corresponding to the formula:



will accelerate the growth of animals when incorporated as an additive in the basal adequate diet in amounts within the range of 0.01 to 0.1%.

Example: The accompanying tables show the results of a series of experiments in which Arbor-Acres-Vantress-Cross chickens were used. This is a well known type of so-called broiler chicken. A number of day-old chicks were divided into groups of 25 each. The groups were raised as separate groups under the same conditions and environment, except that the control group did not receive any diamino diphenyl arsinic acid in their diet. The tables on the following page show the results of this study.

## Growth-Promoting Chemical Additives

| Group          | No. of Birds | Average Weight (gm.) |           | Weight Percent Over Control | Feed Consumption (lb.) | Feed Efficiency |
|----------------|--------------|----------------------|-----------|-----------------------------|------------------------|-----------------|
|                |              | Day-old              | 4-wk.-old |                             |                        |                 |
| Control.....   | 25           | 38                   | 499       | -----                       | 2.06                   | 2.03            |
| DDAA, 0.05%--- | 25           | 38                   | 555       | 11                          | 2.20                   | 1.93            |
| DDAA, 0.1%---  | 25           | 38                   | 565       | 13                          | 2.20                   | 1.94            |

| Group          | Av. Weight at 4-wk.-old (gm.) | Av. Weight at 6-wk.-old (gm.) | Av. Weight at 7-wk.-old (gm.) | Feed              |            |
|----------------|-------------------------------|-------------------------------|-------------------------------|-------------------|------------|
|                |                               |                               |                               | Consumption (lb.) | Efficiency |
| Control.....   | 499                           | 855                           | 1,101                         | 5.60              | 2.39       |
| DDAA, 0.05%--- | 555                           | 951                           | 1,214                         | 5.87              | 2.27       |
| DDAA, 0.1%---  | 565                           | 919                           | 1,160                         | 5.72              | 2.33       |

The use of diamino diphenyl arsinic acid as an additive in the diet of swine to promote growth is illustrated in a series of tests now to be described. Four litters numbering 30 pigs were used for the test. These were crossbred litters. Their sows were each a Berkshire-Poland China cross and each was bred to a purebred Hampshire boar. Prior to weaning, all of the pigs were treated and handled in the same way. The males were castrated at 34 days. The same prestarter feed was available to all pigs up to the time of weaning.

At the time of weaning at 6 weeks and 6 days of age, the 30 pigs were randomly divided by sex, weight, and breeding into three groups of ten to a group. The three groups are designated as Groups A, B, and C. Each group of ten was placed in a separate but similar concrete confinement pen, each provided with an automatic feeder and an automatic waterer. Each group was fed the same feed and in all ways treated in the same manner except that Group A (here called the control group) was fed a basal ration that did not contain any diamino diphenyl arsinic acid.

The ingredients of the basal pig ration at different pig weights was as follows:

| Ingredients           | 30 to 110 lbs. | 110 to 160 lbs. | 160 lbs. to Market Weight |
|-----------------------|----------------|-----------------|---------------------------|
| Ground yellow corn    | 1,300          | 1,450           | 1,600                     |
| 35% supplement        | 600            | 450             | 300                       |
| Molasses              | 90             | 90              | 90                        |
| Salt                  | 10             | 10              | 10                        |
| Est. protein, percent | 16.2           | 14.2            | 12.2                      |

The test time was divided into three periods of 30 days (First Period), 30 days (Second Period), and 48 days (Third Period), respectively, making a total of 108 days. The table on the following page sets forth the results for the three periods and the 108 day summary.

Summarizing the entire 108 day test period, Group B whose basal feed ration contained 0.0125% diamino diphenyl arsinic showed a daily weight gain of 5.1% and an improved



## Growth-Promoting Chemical Additives

feed efficiency of 6.2% over the control Group A. Group C, whose basal feed ration contained 0.025% diamino diphenyl arsinic acid, showed an increased daily weight gain of 8.2% and an improved feed efficiency of 6.2% over the control Group A.

|                                | Group A<br>Control | Group B<br>0.0125%<br>DDAA | Group C<br>0.025%<br>DDAA |
|--------------------------------|--------------------|----------------------------|---------------------------|
| <b>First Period, 30 days:</b>  |                    |                            |                           |
| No. Animals.....               | 10                 | 10                         | 10                        |
| Av. Init. Wt., lbs.....        | 29.3               | 29.0                       | 29.3                      |
| Av. Final Wt., lbs.....        | 63.2               | 60.5                       | 65.9                      |
| Av. Da. Gain, lbs.....         | 1.13               | 1.05                       | 1.22                      |
| Feed/lb. Gain, lbs.....        | 2.61               | 2.60                       | 2.58                      |
| <b>Second Period, 30 days:</b> |                    |                            |                           |
| No. Animals.....               | 10                 | 10                         | 10                        |
| Av. Init. Wt., lbs.....        | 63.2               | 60.5                       | 65.9                      |
| Av. Final Wt., lbs.....        | 107.4              | 107.9                      | 112.5                     |
| Av. Da. Gain, lbs.....         | 1.47               | 1.58                       | 1.55                      |
| Feed/lb. Gain, lbs.....        | 3.88               | 3.16                       | 3.65                      |
| <b>Third Period, 48 days:</b>  |                    |                            |                           |
| No. Animals.....               | 10                 | 10                         | 10                        |
| Av. Init. Wt., lbs.....        | 107.4              | 107.9                      | 112.5                     |
| Av. Final Wt., lbs.....        | 119.8              | 208.2                      | 213.5                     |
| Av. Da. Gain, lbs.....         | 1.93               | 2.09                       | 2.10                      |
| Feed/lb. Gain, lbs.....        | 4.27               | 4.11                       | 4.07                      |
| <b>Overall, 108 days:</b>      |                    |                            |                           |
| No. Animals.....               | 10                 | 10                         | 10                        |
| Av. Init. Wt., lbs.....        | 29.3               | 29.0                       | 29.3                      |
| Av. Final Wt., lbs.....        | 190.8              | 208.2                      | 213.5                     |
| Av. Da. Gain, lbs.....         | 1.58               | 1.66                       | 1.71                      |
| Feed/lb. Gain, lbs.....        | 3.84               | 5.60                       | 3.65                      |

### Beta-Nitropropionic Acid

J. Kamlet (U.S. Patent 3,157,511; November 17, 1964; assigned to Celanese Corporation of America) found that the addition of beta-nitropropionic acid, or a salt thereof, in minor amounts to a poultry feed greatly increases the efficiency with which this feed is utilized by the poultry and greatly increases the rate of growth of the poultry.

**Example 1:** To illustrate the growth-stimulating effects and the increase in feed utilization obtained with the use of beta-nitropropionic acid in poultry feeds, day-old chicks were fed on a basal diet. Duplicate pens of 12 New Hampshire chicks per pen were fed this basal diet for 28 days. Beta-nitropropionic acid and other antibiotics, as well as beta-nitropropionic acid in conjunction with other antibiotics, were fed to similar duplicate pens of 12 New Hampshire chicks per pen for 28 days. The following results were obtained.

| <u>Supplement</u>   | <u>Fed to:</u> |
|---|----------------|
| Control (none)  | Pen A          |
| 5 g. beta-nitropropionic acid/ton                                 | Pen B          |
| 10 g. beta-nitropropionic acid/ton                                | Pen C          |
| 20 g. beta-nitropropionic acid/ton                                | Pen D          |
| 50 g. beta-nitropropionic acid/ton                                | Pen E          |
| 10 g. procaine-penicillin/ton                                     | Pen F          |
| 10 g. procaine-penicillin plus 20 g. beta-nitropropionic acid/ton | Pen G          |
| 10 g. zinc bacitracin/ton   | Pen H          |
| 10 g. zinc bacitracin plus 20 g. beta-nitropropionic acid/ton     | Pen I          |
| 10 g. aureomycin/ton  | Pen J          |
| 10 g. aureomycin plus 20 g. beta-nitropropionic acid/ton          | Pen K          |

The average weight and feed conversions per pen are shown on the tables on the following page.

## Growth-Promoting Chemical Additives

AVERAGE WEIGHT

|            | First Rep. | Second Rep. | Average Rep. | Percent Response |
|------------|------------|-------------|--------------|------------------|
| Pen A..... | 248.4      | 251.4       | 249.9        | -----            |
| Pen B..... | 261.2      | 269.4       | 265.3        | 6.2              |
| Pen C..... | 287.0      | 279.6       | 283.3        | 13.4             |
| Pen D..... | 299.8      | 307.7       | 303.9        | 21.6             |
| Pen E..... | 301.7      | 296.4       | 303.6        | 21.5             |
| Pen F..... | 288.6      | 279.4       | 284.0        | 13.6             |
| Pen G..... | 312.6      | 310.8       | 311.7        | 24.7             |
| Pen H..... | 281.6      | 290.6       | 286.1        | 14.5             |
| Pen I..... | 301.6      | 304.4       | 303.0        | 21.2             |
| Pen J..... | 274.4      | 279.6       | 277.0        | 10.8             |
| Pen K..... | 310.4      | 312.8       | 311.6        | 24.7             |

FEED CONVERSION

|            | 2.70 | 2.84 | 2.77 | ----- |
|------------|------|------|------|-------|
| Pen A..... | 2.62 | 2.58 | 2.60 | 6.1   |
| Pen B..... | 2.51 | 2.48 | 2.50 | 9.7   |
| Pen C..... | 2.49 | 2.50 | 2.50 | 9.7   |
| Pen D..... | 2.46 | 2.50 | 2.48 | 10.5  |
| Pen E..... | 2.61 | 2.58 | 2.60 | 6.1   |
| Pen F..... | 2.40 | 2.38 | 2.39 | 13.7  |
| Pen G..... | 2.58 | 2.49 | 2.54 | 8.3   |
| Pen H..... | 2.47 | 2.40 | 2.44 | 11.9  |
| Pen I..... | 2.61 | 2.51 | 2.56 | 7.6   |
| Pen J..... | 2.44 | 2.39 | 2.42 | 12.6  |
| Pen K..... |      |      |      |       |

It will thus be noted that:

- (a) The addition of from 5 to 50 grams of beta-nitropropionic acid per ton of feed, produces a 6.2 to 21.6% increase in weight over that obtained without such additions to the basal ration;
- (b) The addition of beta-nitropropionic acid to poultry feeds containing antibiotics considerably enhances the weight increases obtained. Thus, adding 10 grams (per ton of feed) of procaine-penicillin, of zinc bacitracin and of aureomycin produces weight increases of from 10.8 to 14.5%. The addition of 10 grams of antibiotic and 20 grams of beta-nitropropionic acid (per ton of feed) produces weight increases of 21.2 to 24.7%.
- (c) Beta-nitropropionic acid alone, added in amounts of from 5 to 50 grams per ton of feed, improves feed conversion from 6.1 to 10.5%. Beta-nitropropionic acid (20 g./ton of feed) improves feed conversion from 6.1 to 13.7%. The beta-nitropropionic acid may thus be said to possess an antibiotic-extending or antibiotic-sparing function in poultry feeds, or may be considered as having a synergistic activity in promoting said activity.

The alkali metal salts and the alkaline earth metal salts of the beta-nitropropionic acid are as effective as the free acid for improving the feed utilization and increasing the weight of poultry fed with a basal diet containing the same. In preparing the poultry feeds, the beta-nitropropionic acid or an alkali metal or alkaline earth metal salt thereof, may be added directly to the feed, in a solid form or in solution. Alternatively, these substances may be intimately mixed with another feed ingredient, such as one of the commonly used feed ingredients like bone meal, calcium carbonate, etc., to produce a premix, e.g., a 5 to 10% premix in bone meal. An appropriate amount of this premix is then distributed throughout the entire feed in a feed mixer.

Example 2: Tests were conducted on five lots of six pigs per lot for 112 days (16 weeks). The pigs were fed a basic ration to which was added varying amounts of beta-nitropropionic



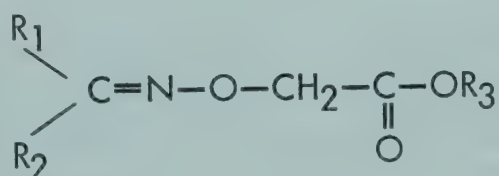
## Growth-Promoting Chemical Additives

acid. The following results were obtained.

| Lot No. | Grams of beta-nitro-propionic acid added per ton of feed | Average Initial Weight (lbs.) | Average Final Weight (lbs.) | Average Daily Gain (lbs.) | Average Feed Consumed (lbs.) | Average Feed (lbs.) per lb. gain |
|---------|--|-------------------------------|-----------------------------|---------------------------|------------------------------|----------------------------------|
| 1-----  | 0  | 25.8                          | 170.5                       | 1.29                      | 492                          | 3.40                             |
| 2-----  | 5  | 25.6                          | 175.0                       | 1.33                      | 488                          | 3.27                             |
| 3-----  | 10   | 26.1                          | 190.0                       | 1.43                      | 480                          | 2.99                             |
| 4-----  | 25   | 26.0                          | 204.0                       | 1.59                      | 611                          | 3.00                             |
| 5-----  | 50   | 25.7                          | 200.5                       | 1.56                      | 614                          | 3.06                             |

### N-Substituted Aminooxyacetic Acid

The addition of a small amount of a N-substituted aminooxyacetic acid compound (including salts and esters) to livestock and poultry feed results in improved growth and feed efficiency as described by H.R. Rosenberg (U.S. Patent 3,284,210; November 8, 1966; assigned to E.I. du Pont de Nemours and Company). Useful compounds according to this process are those of the following formula:



where:  $R_1$  is hydrogen or alkyl of 1 through 6 carbon atoms;  
 $R_2$  is hydrogen, alkyl of 1 through 5 carbon atoms or the radical  $-\text{CO}_2R_3$ ;  
 with the proviso that  $R_1$  and  $R_2$  can be joined becoming  $-(\text{CH}_2)_n-$   
 where  $n$  equals 4, 5 or 6 and a methyl, ethyl, propyl or isopropyl group  
 can be substituted on the ring; and  
 $R_3$  is hydrogen, alkyl of 1 through 4 carbon atoms, sodium, potassium, calcium or magnesium.

It should be understood that the ring can be substituted with one, two or three alkyl radicals each having up to 3 carbon atoms without departing from the scope of this process. Preferred compounds are those where  $R_1$  and  $R_2$  are joined to form cycloalkylidene derivatives without alkyl substituents on the ring.

The active compound is mixed with feed by any conventional means. The amount added to the animal diet will depend upon the attendant circumstances and the nature of the effect desired. The level of the compound in the feed should not be less than 0.0005% nor greater than about 0.5%, based on the total weight of the animal's feed. Expressed in another way, the amount of active compound in the animal's feed will ordinarily be within the range from about 4 to 5,000 grams per ton of animal feed.

Example 1: Two groups of day-old New Hampshire chicks are placed in separate pens and fed under controlled diet conditions for a period of 28 days. One group is provided with a basal chick diet while the second group is fed with same diet to each ton of which is added 75 grams of cyclohexylidene aminooxyacetic acid. The group receiving the cyclohexylidene

## Growth-Promoting Chemical Additives

aminoxyacetic acid supplement shows an average improvement of 24.5% in weight increase compared with the control group. Also, the group receiving the feed supplement of this process exhibits a significant improvement in feed conversion of approximately 10.0% when compared with the control group.

Example 2: A test is conducted using three identical groups of 40 cross-bred male chicks. Group 1 is fed a basal diet only. Group 2 is fed a basal diet plus 0.01% cyclohexylidene aminoxyacetic acid. Group 3 is fed a basal diet plus 0.01% isopropylidene aminoxyacetic acid. After a four-week floor pen test the results obtained are as follows:

| Group  | Treatment   | Index of performance (gain <sup>2</sup> /feed) | Average weight gain | Average feed/gain |
|--------|---|--|---------------------|-------------------|
| 1----- | Basal only  | *315 (0)                                       | 517                 | 1.64              |
| 2----- | Basal plus 0.01% cyclohexylideneaminoxyacetic acid. | *343 (9)                                       | 548                 | 1.60              |
| 3----- | Basal plus 0.01% isopropylideneaminoxyacetic acid.  | *339 (8)                                       | 545                 | 1.61              |

\*Number in parentheses indicate percentage increases over basal diet on basis of index of performance.

Example 3: A commercial swine diet (20% protein) was used in this study. In addition to a control group, additional separate groups of swine are fed, besides the basal diet, cyclohexylidene aminoxyacetic acid salt in an amount of, respectively, 10, 25, 50, 75, 100, and 200 grams per ton of feed, with resulting significant improvement in weight increase and feed conversion evidenced in those groups receiving the cyclohexylidene aminoxyacetic acid feed supplement.

### 5-Nitro-2-Furaldehyde-2-(2-Hydroxyethyl)-Semicarbazone

M.F. Paul (U.S. Patent 3,039,875; June 19, 1962; assigned to Norwich Pharmacal Company) found that it is possible to achieve remarkable and surprising stimulation of growth, general improvement in health and appearance, and enhanced feed efficiency in animals through the administration of a supplemented animal feedstuff containing, in addition to the animal's basal ration, a small quantity of 5-nitro-2-furaldehyde-2-(2-hydroxyethyl)-semicarbazone.

The animal feed serves as a carrier for a small quantity of 5-nitro-2-furaldehyde-2-(2-hydroxyethyl)-semicarbazone which is mixed with it. Its preparation may be readily accomplished through any of the common methods employed for dispersing supplementary agents uniformly in feed, such as tumbling, grinding, and stirring. The amount of 5-nitro-2-furaldehyde-2-(2-hydroxyethyl)-semicarbazone added to the basal diet may be varied within limits. It is usually sufficient to add an amount within the range of about 10 to 100 mg. per kilogram of ration to achieve optimum results, the preferred level being about 20 to 50 mg. per kilogram. The 50 mg. per kilogram level is particularly operable in certain species, e.g., swine, while a level of about 20 mg. per kilogram is desirable in others, e.g., chickens. Tests on chickens showed a surprising weight gain and feed conversion.

Example 1: Groups of 50, day-old broiler chickens were segregated to afford a control group receiving the basal diet for comparison with other groups to which the basal diet containing varied amounts of the compound, 5-nitro-2-furaldehyde-2-(2-hydroxyethyl)-semicarbazone,



## Growth-Promoting Chemical Additives

was offered, and to permit comparison of the effects produced by varied amounts of that compound. Each group received in its ration nitrofurazone, in the amount of 0.0055%. The results of such a test, representing a total of 150 birds for each group maintained in the test for 63 days, are shown in the following table.

| <u>Group</u>     | <u>Amount of Compound, (grams/ton)</u> | <u>Average Weight, (Grams)</u> |               |             | <u>Feed Efficiency, Grams Feed Per Grams Gain in Weight</u> |
|------------------|--|--------------------------------|---------------|-------------|---|
|                  |  | <u>Male</u>                    | <u>Female</u> | <u>Avg.</u> |   |
| Basal            |  | 1,396                          | 1,089         | 1,243       | 2.71  |
| Basal + Compound | 5                                      | 1,397                          | 1,187         | 1,292       | 2.60  |
| Basal + Compound | 10                                     | 1,441                          | 1,225         | 1,333       | 2.59  |
| Basal + Compound | 15                                     | 1,443                          | 1,185         | 1,314       | 2.60  |
| Basal + Compound | 20                                     | 1,468                          | 1,181         | 1,325       | 2.56  |

Example 2: This test was conducted as above except that the feed offered to the groups did not contain nitrofurazone. The results are shown below.

| <u>Group</u>     | <u>Amount of Compound, (grams/ton)</u> | <u>Average Weight, (grams)</u> | <u>Feed Efficiency, Grams Feed Per Grams Gain in Weight</u> |
|------------------|--|--------------------------------|---|
| Basal            |  | 1,341                          | 2.86  |
| Basal + Compound | 10                                     | 1,381                          | 2.78  |
| Basal + Compound | 25                                     | 1,393                          | 2.77  |

### Carboxymethoxylamine

J. Kamlet (U.S. Patent 3,051,573; August 28, 1962; assigned to E.I. du Pont de Nemours and Company) found that the addition of a small amount of carboxymethoxylamine to livestock and poultry rations results in improved animal growth and better feed efficiency. The inclusion of this compound in conventional poultry feeds and diets has a particularly stimulating effect on chick growth. As used here, carboxymethoxylamine is intended to include closely related derivative compounds including salts and alkali metal salts. A particularly preferred compound is carboxymethoxylamine hemi-hydrochloride.

The amount of carboxymethoxylamine added to the animal diet will depend upon the attendant circumstances and the nature of the effect desired. The level of the compound in the feed should not be less than 0.001% nor greater than about 0.1%, based on the total weight of the animal's feed. Expressed in another way, the amount of carboxymethoxylamine in the animal's feed will ordinarily be within the range from about 10 to 1,000 grams of carboxymethoxylamine per ton of animal feed.

Using the hemi-hydrochloride compound, for example, a preferred amount of from 20 to 200 grams of this compound per ton of feed exerts a growth improvement in feeding chicks approximately equal to that realized by feeding chicks procaine penicillin at the level which is commercially practiced.

## Growth-Promoting Chemical Additives

Example: Eight groups each containing 48 male cross-breed chicks are used in this test. Results after a period of only two weeks are as follows.

| Group  | Diet                       | Weight<br>Gain,<br>Average | Feed/Gain,<br>Average |
|--------|----------------------------|----------------------------|-----------------------|
| A..... | Basal only.....            | 152                        | 1.60                  |
| B..... | Basal only.....            |                            |                       |
| C..... | Basal plus 0.002% CMA..... | 166                        | 1.47                  |
| D..... | Basal plus 0.002% CMA..... |                            |                       |
| E..... | Basal plus 0.01% CMA.....  | 185                        | 1.45                  |
| F..... | Basal plus 0.01% CMA.....  |                            |                       |
| G..... | Basal plus 0.02% CMA.....  | 185                        | 1.44                  |
| H..... | Basal plus 0.02% CMA.....  |                            |                       |



## POULTRY FEEDS

### GROWTH ENHANCING ADDITIVES FOR POULTRY FEED

Much has been learned about the nutritional requirements of poultry, which has led to the development of complete scientific rations. Success has been had with certain substances such as antibiotics which act as growth promoters, even though they have no food value of themselves. These processes deal with upgrading poultry food by specific additives and means so as to realize still further weight gains and improvements in feed efficiency.

#### Diastatic Malt to Upgrade Barley in Feeds

C.M. Hollenbeck (U.S. Patent 2,988,449; June 13, 1961; assigned to Wisconsin Malting Co.) found that diastatic barley malt effectively up-grades the metabolizable value of barley in poultry feed and that this up-grading process is dependent upon the barley gum degrading enzyme content of the malt, i.e., the cytolytic enzymes in the malt (often referred to as cytases, gumases or beta-polyglucosidases).

Examples of the relative enzyme activities and barley up-grading efficiencies in feeds of 5 different samples of malt are shown in Table 1. The data show that the growth efficiency does not correlate with alpha-amylase or with total amylase activity (diastatic power), but does correlate with cytolytic activity.

TABLE 1

| Malt Sample | Relative Enzyme Activity of Malt Samples <sup>1</sup> |   |  |   |
|-------------|---|---|--|---|
|             | Diastatic <sup>2</sup><br>Activity,<br>(Percent)      | Alpha <sup>3</sup><br>Amylase<br>Activity,<br>(Percent) | Cytolytic <sup>3</sup><br>Activity,<br>(Percent) | Feed <sup>4</sup><br>Efficiency,<br>(Percent) |
| 1.....      | 80  | 71  | 100  | 100   |
| 2.....      | 75  | 100   | 100  | 100   |
| 3.....      | 100   | 86  | 92   | 70  |
| 4.....      | 34  | 53  | 72   | 50  |
| 5.....      | 10  | 25  | 88   | 60  |
| 6.....      | 76  | 80  | 85   | 60  |

<sup>1</sup> Relative activities based on the most active at 100%.

<sup>2</sup> Cereal Laboratory Methods, fifth edition, pp. 94-98.

<sup>3</sup> Twenty four grams of ground barley and 1 g. of ground malt are mashed at 38° C. in 200 ml. of water. Replicate samples are started at spaced time intervals. The reaction is stopped at predetermined time intervals by the addition of 25 ml. of 40% trichloroacetic acid solution. The mixtures are then heated to 72° C., with stirring, dispersed while hot in a Waring Blendor (3 minutes) and then cooled in running cold water. The cold suspension is centrifuged, the supernatant decanted thru cheese cloth, and the viscosity of the clear extracts determined. The relative rate of viscosity lowering is a measure of cytolytic activity.

<sup>4</sup> Calculated using average weekly growth gains and based on the largest gain being 100%.

## Poultry Feeds

The actual growth response of chickens on rations comprising 60% barley with and without various levels of malt are shown in Table 2.

**TABLE 2: CHICK GROWTH RESPONSE WITH VARIOUS LEVELS OF MALT**

| Malt Level (Percent Total Ration) <sup>1</sup> | Average Weight in Grams (20 Chicks per group)—Weeks Age |     |     |
|--|---|-----|-----|
|  | 2   | 4   | 6   |
| 0.0.....                                       | 120   | 314 | 576 |
| 0.6.....                                       | 128   | 331 | 620 |
| 1.2.....                                       | 129   | 337 | 647 |
| 2.4.....                                       | 135   | 340 | 662 |
| 4.8.....                                       | 143   | 338 | 665 |

<sup>1</sup> Ration comprised 40% basal mixture of alfalfa leaf meal, soya bean meal, bone meal, granite grits, limestone, iodized salt, fish meal and a vitamin mineral mixture and 60% barley plus malt adjunct.

One of the major factors determining the value of malt for this use is its cytolytic activity. This depends upon several things, among which is the type of barley used in the manufacture of the malt. Midwestern, 6-row white and Canadian, 6-row, blue barleys appear to produce malt with higher cytolytic activity.

### Diastatic Barley Malt Plus Fungal Enzyme

In another process C.M. Hollenbeck (U.S. Patent 2,988,448; June 13, 1961; assigned to Wisconsin Malting Co.) found that diastatic barley malt and fungal enzyme preparations act synergistically in increasing the value of barley and other fibrous grains in poultry feed. An example of the synergistic effect of the combination of malt enzymes and fungal derived enzymes in increasing the growth rate of chickens on rations containing barley is given in the following table.

**Comparison of Chick Weights After Being Fed Barley Based Rations Supplemented with Various Enzymes and Combinations Thereof**

| Test No. | Supplementary Enzyme                         | Relative Enzyme Potency of Supplement (Alpha-Amylase Units) | Average Weight of Chicks (grams) (20 chicks per group) |          |
|----------|--|---|--|----------|
|          |  |   | 4 Weeks  | 10 Weeks |
| 1.....   | Control (no enzyme).....                     |   |  |          |
| 2.....   | Malt.....                                    |   | 292  | 1,195    |
| 3.....   | Fungal Preparation (crude).....              | 50  | 319  | 1,263    |
| 4.....   | Malt plus Fungal preparation (crude).....    | 194   | 334  | 1,308    |
|          |  | 140   | 363  | 1,370    |
| 5.....   | Malt plus Fungal preparation (purified)..... | 194   | 348  | 1,434    |

The barley based ration comprised 40% basal mixture of alfalfa leaf meal, soya bean meal, bone meal, granite grits, limestone, iodized salt, fish meal and a vitamin mineral mixture and 60% barley plus enzyme supplement. The enzyme supplement amounted to about 1.5% by weight of the barley based ration, with the exception of the malt alone, which was 4%



by weight of the barley based ration. The fungal enzyme supplementary preparation used in the above tests was "mold bran" or a dried material resulting from the growth of Aspergillus oryzae on steamed wheat bran.

In test No. 4 the ground mold bran was mixed with an equal weight of ground, diastatic malt. In test No. 5 the fungal material was a purified preparation called Rhozome S. The fungal enzyme preparation was added to ground, diastatic malt at the level of 5% of the enzyme mixture.

### Growth Factors from Fermentation of Wet Wheat Bran

C.M. Ely, A.P. De Luca, and F. Andriuli (U.S. Patent 3,151,983; October 6, 1964; assigned to Nopco Chemical Company) have developed a process for increasing the growth response and feed efficiency of poultry, by introducing into a poultry feed which is fed to poultry, a feed supplement comprising a mixture of (a) a material prepared by fermenting wet wheat bran under aerobic conditions, using Aspergillus oryzae and (b) a material prepared by fermenting wet wheat bran under aerobic conditions using Bacillus subtilis.

Example 1 — Preparation: Material A can be prepared in the following manner. Wheat bran is added to a large horizontal cooker having a tight fitted cover. The wheat bran is steamed under a pressure of from about 0.5 to 1.0 psig so that complete pasteurization is effected and partial sterilization takes place. The steam is then released from the cooker and the bran is allowed to cool to about 55°C. at which point, tap water is sprayed onto the bran for cooling and wetting purposes. The wheat bran is then inoculated with a spore slurry containing Aspergillus oryzae. During these operations, aseptic techniques are used. The inoculated wet bran mixture is then placed on trays. These trays are placed in sterilized incubator rooms where the temperature and relative humidity are carefully controlled. The temperature of the bran mixture on the trays is maintained at from about 25° to 40°C. by circulating filtered air through the incubator rooms.

The relative humidity of the rooms is maintained at at least 95% by bleeding low pressure steam into the room. The fermentation is allowed to proceed for from about 40 to 75 hours under the above controlled conditions. The wet, partially fermented wheat bran is then removed from the trays, again placed in the horizontal cooker and water sprayed on the partially fermented bran to rewet same. Rewetting results in further fermentation of the wet inoculated wheat bran.

The inoculated rewet bran is allowed to ferment for about 40 to about 60 additional hours under the same controlled temperature and humidity as aforescribed. Finally, warm dry air, at a temperature of from about 30° to 55°C. is circulated through the incubator rooms until the fermented bran is dry.

Material B is prepared in the following manner. Wheat bran is added to a horizontal cooker with a tight fitting cover and then steamed under a pressure of from 0.5 to 1.0 pound per square inch gauge. The steam is then released and the bran is cooled to about 55°C. at which time water is sprayed onto the bran for cooling and wetting purposes. The wheat bran is then inoculated with Bacillus subtilis. During all these operations, aseptic techniques are used in order to avoid contamination. The inoculated bran is then placed in sterilized

## Poultry Feeds

trays in a sterilized room under controlled temperature and relative humidity. The temperature of the bran is maintained at from about 25° to 45°C. by circulating filtered air through the incubator rooms. The relative humidity of the room is maintained at at least 95% by bleeding low pressure steam into the room. The bacterial fermentation is conducted for from about 30 to 60 hours under the above controlled conditions. Air, at a temperature of from about 30° to 55°C. is then circulated through the room until the wheat bran ferment is air dry.

During fermentation of Material A and Material B, considerable heat is developed. This is dissipated by circulating air through the fermentation room. As the fermentation decreases in activity, less heat is developed, and less air is circulated. When there is no longer need for air, fermentation is considered complete.

The manner of wetting the bran is not critical and so long as there is present from about 40 to about 65% of water based on the total weight of the water plus the wheat bran.

**Example 2 — Use:** The general procedure employed was as follows: All tests were conducted on one day old White Vantress cockerels. The test groups varied from 14 to 30 chicks and were divided into three equal lots of chicks. All experiments reported were of 38 to 43 days duration. During this time, the chicks were housed in electrically heated starting batteries and supplied with food and water ad libidum. The temperature of the starting batteries was maintained from about 80° to 85°F. A special effort was made to minimize the effect of cage position upon the biological response by placing different lots of the same group in different cage positions. All chicks and feed were weighed at the beginning of the experiment and all pertinent data was recorded at weekly intervals.

Effect of Feeding Basal Rations Supplemented With Material A,  
Material B and Mixtures Thereof, on the Growth Response and  
Feed Efficiency of Poultry

|                                 | Group                           | No.<br>Chicks | Amount of<br>A and/or<br>B Added<br>(Lbs./Ton<br>of Feed) | Average<br>Gain/Bird<br>(Grams) | Improve-<br>ment in<br>Gain<br>Compared<br>to Control<br>(Percent) | Feed-Gain<br>Ratio | Improve-<br>ment in<br>Feed-Gain<br>Compared<br>to Control<br>(Percent) |
|---------------------------------|---------------------------------|---------------|---|---------------------------------|--|--------------------|---|
| Example I.<br>28 Day Test.....  | Control.....                    | 30            |   | 383.8                           |  | 2.255              |   |
|                                 | Material A plus Material B..... | 30            | 2   | 430.6                           | 12.2   | 2.073              | 8.1   |
|                                 |                                 |               | 2   |                                 |  |                    |   |
| Example II.<br>38 Day Test..... | Control.....                    | 16            |   | 658.0                           |  | 2.257              |   |
|                                 | Material B.....                 | 16            | 8   | 733.2                           | 11.4   | 2.206              | 2.5   |
|                                 | Material A.....                 | 15            | 8   | 736.9                           | 12.0   | 2.165              | 4.1   |
|                                 | Material A plus Material B..... | 14            | 4   | 803.0                           | 22.0   | 2.105              | 6.7   |
|                                 |                                 |               | 4   |                                 |  |                    |   |

Either Material A or Material B alone, when added to a basal ration and fed to poultry, improve the growth response and feed efficiency of poultry. When Material A and Material B are combined and the combination added to a basal ration and fed to poultry, a growth response and feed efficiency are obtained which is greater than the growth response and feed efficiency obtained by feeding to poultry a basal ration supplemented with either



## Poultry Feeds

Material A alone or Material B alone. Separate portions of Material A and separate portions of Material B were individually washed with approximately 16.5 pounds of water for each pound of Material A, and 16.5 pounds of water for each pound of Material B, thereby extracting substantially all of the enzymes. When substantially all of the enzymes were extracted from Material B, the growth response remained high especially when compared to the amount of enzymes remaining therein. However, the growth response obtained using washed Material A was negligible.

### Growth Promoter from Dried Distillers Solubles

J.R. Couch and H.D. Stelzner (U.S. Patent 3,119,692; January 28, 1964) have a process for preparing growth promoting concentrates.

Example 1 — Preparation of S-300: To fifty pounds of dried distillers solubles (a composite sample prepared by the Distillers Feed Research Council of distillers solubles from various companies) was added 16 liters of 6 N sulfuric acid, and the mixture stirred to moisten all of the material. The mixture was then autoclaved for 30 minutes at 15 lbs./sq. in. pressure at 121°C. in a conventional steam-sterilizer autoclave. After cooling, a saturated solution of sodium hydroxide in water was slowly added to bring the mass to a pH of 7; about a gallon of alcohol also being added to facilitate the mixing.

Eighty liters of 95% ethyl alcohol was then added to the mixture and vigorously stirred with a propellor type mixer for about 6 to 8 hours. The resulting mixture was then filtered and the residue reextracted with about 50 liters of 80% ethyl alcohol. The mixture was then filtered and the alcoholic filtrates combined and evaporated under vacuum at a temperature of about 75° to 80°C. The evaporation was continued until the volume of the residue was about 5 liters. This residue, in the form of a thick dark brown tar, was then extracted with about 20 liters of acetone. The acetone was thoroughly mixed with the residue and the resulting suspension was allowed to stand for about 18 hours. The supernatant aqueous acetone solution was separated from the residue and then filtered.

The residue was then thoroughly mixed with 20 liters of 80% acetone-water and again allowed to stand for about 18 hours. The supernatant acetone layer was syphoned off, filtered and combined with the first acetone extracts. The residue was again resuspended with 20 liters of 80% acetone-water and the resulting suspension allowed to stand for about 18 hours. The acetone layer was separated and combined with the previous acetone extracts for concentration. The combined acetone extracts were evaporated under diminished pressure at a maximum temperature of about 80°C. to a concentrate of about one liter.

The resulting concentrate was then transferred to a second vessel; a small amount of ethyl ether being used to wash out the first vessel. The residue remaining in the first vessel was mixed with 100 to 200 ml. of acetone and the resulting acetone layer separated. This extraction with acetone was repeated twice more giving a total of about 500 ml. of acetone extracts. The extracted gummy mass is then discarded.

To the main portion of the one liter concentrate in the second vessel was then added about 8 liters of ethyl ether and the mixture stirred for sufficient time to insure thorough mixing of the material. After settling for about 12 hours, the ether solution was separated from the



residue. The ether solution was then washed with 500 ml. of distilled water and the recovered aqueous phase was concentrated under vacuum at a maximum temperature of 70°C. The washed ether solution was discarded. The concentrated aqueous extract was washed twice with 100 to 200 ml. of acetone and the resulting acetone extract and the acetone extract from the residue in the first vessel were combined with the residue obtained from the ethyl ether extraction. Sufficient acetone was then added to make a total of 8 liters of acetone. The acetone suspension was stirred and the resulting mixed suspension placed in the cold room and allowed to stand for about 18 hours.

The acetone layer was separated from the gummy residue and the gummy residue washed twice with one-half liter portions of acetone which were added to the main acetone extracts. The acetone insoluble residue was discarded. The acetone extracts were then combined and evaporated in vacuum at a temperature of 70°C. to obtain about 50 to 75 ml. of gummy residue. To this gum was added 500 ml. of isopropanol:chloroform (75:25 v./v.) and the mixture was shaken to dissolve the gum. The resulting solution was then placed in a cold chest for about 48 hours. During this period a precipitate formed and was recovered by centrifugation in a refrigerated centrifuge at 2,500 rpm for 45 minutes at a temperature of -20°C.

The precipitate consisting of about 800 mg. of material was suspended in 50 ml. of water and the resulting suspension filtered to remove insoluble material. The water solution containing the active component was evaporated in a rotary flask evaporator to dryness. To this dry residue was then added 100 ml. of acetone. After thorough mixing, the acetone suspension was filtered and evaporated to dryness. To the dry residue was then added 100 ml. of isopropanol:chloroform (75:25) and the resulting solution was again placed in the cold chest for 48 hours. The solution at this point contained a white precipitate which was recovered by centrifugation in a refrigerated centrifuge.

The dried precipitate so obtained was recrystallized from a small amount of water to obtain needles of the microcrystalline class which were found to turn brown at about 290°C. and to char at about 300°C. This product is called S-300. When dried brewers' yeast, condensed fish solubles or dried antibiotic fermentation residue are used in place of the dried distillers solubles, a similar product having enhanced poultry growth promoting activity was obtained.

**Example 2 — Use:** Tests with turkey poults demonstrated the activity of the composition S-300 obtained in accordance with the procedures described in Example 1. In these tests day-old Broad Breasted Bronze poults obtained from a commercial source were used. The dams had received a practical breeder diet presumed to be adequate in all known required nutrients. The poults were individually wing-banded and were distributed at random in groups of 11 in electrically heated battery breeders. The individual groups were then maintained on the experimental nutritionally balanced diet having 42.8% starch and 40% soybean protein.

The corn distillers dried solubles were added to the diet at the expense of soybean protein and starch to maintain a constant protein level. The other fractions were merely added to the basal diet in an amount equivalent to that obtainable from 10% of the dried distillers solubles. After 4 weeks the turkey poults were weighed. The results shown in the following table summarize those obtained from 5 replicates, each containing 11 turkey poults. The tabulated data on the following page clearly show the growth promoting activity of S-300 and the fact that this activity is not due to the mineral content of the dried distillers solubles.



## Poultry Feeds

| <u>Diet</u>  | <u>Four Weeks</u> | <u>Percent Response</u> | <u>Percent Mortality</u> | <u>F.E.<sup>1</sup></u> |
|--|-------------------|-------------------------|--------------------------|-------------------------|
| Basal  | 602.2             | --                      | 7.4                      | 1.72                    |
| Basal + 10% DDS <sup>2</sup>                         | 697.8             | 15.9                    | --                       | 1.42                    |
| Basal + 0.21 mg./kg. of S-300 <sup>3</sup> + 10% DDS | 753.9             | 25.2                    | 5.4                      | 1.33                    |
| Basal + Ash of 10% DDS <sup>4</sup>                  | 627.8             | 4.3                     | 5.4                      | 1.65                    |
| Basal + Ash of 10% DDS + 0.21 mg./kg. of S-300       | 729.9             | 21.2                    | 1.8                      | 1.34                    |
| Basal + 0.21 mg./kg. of S-300 <sup>3</sup>           | 749.3             | 24.4                    | 5.4                      | 1.31                    |
| Basal + Ash of S-300 <sup>4</sup>                    | 618.3             | 2.7                     | 3.6                      | 1.60                    |
| Basal + 1.05 mg./kg. of S-300 <sup>5</sup>           | 752.7             | 25.0                    | 3.6                      | 1.29                    |

<sup>1</sup> Feed efficiency =  $\frac{\text{amount of weight gain}}{\text{amount of feed}}$

<sup>2</sup> Dried distillers solubles.

<sup>3</sup> 0.21 mg./kg. of S-300 is amount equivalent to 10% of dried distillers solubles.

<sup>4</sup> Ash of dried distillers solubles fed at 7.74 g./kg. which is equivalent of 10% dried distillers solubles.

<sup>5</sup> Equivalent to 50% dried distillers solubles.

### Diatomite in Feed

The process of J.C. Eshleman (U.S. Patent 3,271,161; September 6, 1966) involves the adding of hydrated amorphous silica, commonly referred to as diatomaceous earth, to poultry feed, such material being taken from the earth, run through a primary dryer, ground, further dried until it contains 5 to 10% moisture, then further ground to the desired range of fineness so that a minimum of 100% will pass through a 20-mesh screen and a maximum of 80% will pass through a 200-mesh screen, such range of fineness and moisture being highly important for the use intended.

Example 1: Tests to determine increased feed efficiency, better egg shells and dryer litter involved the following: 400 leghorn hens and 400 sex link hens were put on regular complete egg mash with 1% diatomite added. For 2 weeks prior the amount of feed these hens were consuming was checked. With the diatomite in the feed, they ate 7% less feed with no reduction in egg production, plus an increase in shell strength.

Example 2: 10 leghorn hens were put in each of two cages; Group A being control cage, and Group B being test cage. Group A was given regular complete egg mash and water free choice. Group B was given regular complete egg mash with 1% diatomite and water free choice. After 35 days' test results in group A were: Number of birds left - 7; Number of eggs laid - 191; Pounds feed eaten - 95; Litter moisture - fair. Results in group B (with diatomite) were: Number of birds left - 10; Number of eggs laid - 192; Pounds of feed

## Poultry Feeds

eaten - 88; Litter moisture - Excellent. Increased feed efficiency, 7% plus. It was also observed that birds in cages had better health (less strain) when feed contained diatomite.

### Bovine Bone Supplement

A poultry feed supplement which acts as an anabolic stimulator and also reduces cannibalism is prepared by the process of E.J. Tucker, Jr. (U.S. Patent 3,256,096; June 14, 1966; assigned to Organic Nutrients, Inc.). In preparing the supplement, freshly butchered bovine bone is employed as the first element processed, the marrow of the bone to provide the fat component of the mixture. The bone may be cut transversely into workable lengths, as say six inch lengths, and then a length may be sliced longitudinally, as by a conventional revolving saw, into strips, as strips averaging approximately one-half inch wide by one-fourth inch thick.

The strips are fed into a cutting machine in which the blades reduce the bone to particle size in cooperation with a screen having perforations therein of approximately one-fourth inch diameter, the screen being of such thickness and the blades being set to revolve in such proximity to the inner face of the screen, that the particles are forced out or extruded through the perforations with limiting dimension being approximately one-fourth inch in diameter and one-half inch in length.

In the process of reducing the bone to particle size any fat, as marrow fat, which has not been wiped off in butchering and slicing or left residually in the cutting machine, is carried through in those bone particles which retain marrow when cut, or which otherwise have marrow thereon by virtue of association with marrow bearing particles during the cutting process.

The bone particles, including the marrow fat therein and carried along therewith, have added thereto a liposopic material, as cottonseed meal, in weight about one-ninth of the weight of the bone particles with marrow fat included, so that the components of the total mixture at this point are in the proportions of liposopic materials 10% to bone and marrow fat 90%. This mixture is now placed in a centrifugal mixer and mixed and stirred until substantial uniformity of admixture is obtained. The bone particles and the liposopic material with the marrow absorbed therein, are then about equal in volume due to the cottonseed meal having absorbed fat and being resultantly swollen.

The mixture of bone particles carrying marrow mixed with the liposopic material is now further mixed with a coating material such as calcium carbonate which is added in weight ratio of from 10% to 20% to the total weight which is thus respectively from approximately 90% to approximately 80% by weight of a final mixture to be hereinbelow described. The coating material and the mixture aforesaid are automatically mixed together, as by a centrifugal mixture or stirrer, and with such homogeneity that each particle is completely coated with a coating of calcium carbonate, calcium carbonate in excess of that comprised in the coating being mixed with the cottonseed meal and marrow fat absorbed therein. Such mixture should thereby occupy in volume a space or volume substantially equal to the space occupied by the bone particles and their calcium carbonate coatings.

When the mixture including the individually coated particles is complete it is placed in



## Poultry Feeds

polyethylene bags which are sealed and thereafter the bags are treated for approximately 30 minutes in a chamber so that pressurized ethylene oxide gas may be forced to penetrate the bag to remain therein to serve as an inhibitor of the growth of bacteria and/or fungus while the bags of poultry food supplement are in storage or while they are being handled prior to being opened and the contents thereof fed to poultry.

Example: 60 young chickens, crosses between White Rock and Rock Cornish were employed. Thirty of these young chickens were placed in one pen and the other thirty were placed in another pen, and the two groups were observed for approximately seven weeks. At the beginning of the period the average weight per chick was less than 5 ounces. One group was fed the bone mixture at the rate of approximately one gram per chick per day throughout the seven-week period while the other group was fed no bone supplement. Both groups, however, were otherwise fed the same amount of conventional types of poultry feed.

At the end of two weeks no evidence of cannibalism was apparent in the bone fed group and the average weight had increased to 8 ounces whereas cannibalism was observed in the group which had not been fed the bone supplement, one chick had died, and the average weight of the remaining chicks was 7.2 ounces. Two weeks later the average weight of the bone fed group had increased to 24.5 ounces and no evidence of cannibalism could be observed; however, one chick in this group died due to other causes.

As for the group not fed the bone supplement, its weight had increased to 22.3 ounces where much evidence of cannibalism was apparent. Later, at the end of the seven week test period, the average weight of the bone fed group was 3 1/2 pounds with no results of cannibalism being observed, whereas at the end of this same period the group which had not been fed the bone supplement averaged 2 pounds, 10 ounces in weight and much evidence of cannibalism was apparent.

### Treated Soybean Oil Meal

A process for treating soybeans which is particularly adaptable for use as a constituent in poultry and livestock feed, which produces a higher fat content meal and changes the molecular structure of the oil, protein and carbohydrates, has been developed by R.W. Lewis (U.S. Patent 3,257,209; June 21, 1966).

In the toasting process, the cracked or flaked soybeans are heated in a steam jacketed toaster to a temperature of approximately 185° to 235°F. for a period of about 80 minutes, the lower temperature range being preferred. The meats are removed from the toaster and immediately placed in a specially designed screw press wherein the oil content is reduced. The mechanical pressure alone is generally sufficient to maintain the elevated temperature of the meats without adding additional heat, however, for best results it is felt that a pressing temperature of about 185° to 235°F. is desirable. The maximum pressure in the press should be between about 3,500 and 5,000 psi. The toasting time and temperature has been found to destroy the enzyme erease factor without being detrimental to the proteins or lecithins present in the meal. The erease factor in the raw soybean has been found to retard the growth of both poultry and livestock.

Example — Feed Preparation: The premix includes the composition shown on the next page.

## Poultry Feeds

| <u>Ingredient</u>                        | <u>Weight, Pounds</u> |
|--|-----------------------|
| Vitamin and mineral concentrate          | 27.0                  |
| Fish, 10% fat, 60% protein               | 96.8                  |
| Meat and bone meal, 10% fat, 60% protein | 87.7                  |
| Calcium                                  | 35.0                  |
| CaH(PO <sub>4</sub> )                    | 35.0                  |
| Salt                                     | 8.7                   |
| Dehydrated alfalfa                       | 114.8                 |

The finished product contains:

| <u>Ingredient</u>        | <u>Weight, Pounds</u> |
|--------------------------|-----------------------|
| Premix                   | 50                    |
| Treated soybean oil meal | 600                   |
| Corn                     | 1,600                 |

The soybean oil meal includes 13% oil and has been initially treated by means of toasting as described above. The above mixture has been found particularly adaptable as a feed for young poultry and livestock, and particularly for turkey poults. In a test wherein the above material is fed to Moorhouse's improved small white turkeys, the birds achieved an average gain in weight of 10 pounds after sixteen weeks as compared to a control group which achieved a 10 pound gain only after twenty weeks. The pounds of feed consumed per bird were substantially identical in the test group as compared to the control group. After a period of twelve weeks, the test group had achieved a weight gain of 6 pounds as compared to only 5 pounds for the control group.

The control group was fed a conventional feed including the ingredients set forth with the exception that the oil from the soybean meal was essentially entirely removed. In addition to the more rapid growth rate, the meat or tissue of the poults fed with the specially treated soybean oil meal had a substantially greater degree of fat distribution or mottling. This feature contributes to a greater degree of tenderness in the finished meat. In addition, the skin of the bird was colored more deeply; this greater degree of pigmentation being due to the xanthophyll present in the retained oil or fat. This enhanced meat product is believed to be obtained through the particular treatment given the soybean oil meal as set forth hereinabove.

### Partial Detoxification of Raw Soybeans

A.C. Groschke (U.S. Patent 3,434,845; assigned to Agway Inc.) found that if the ground raw soybeans are first blended with certain aliphatic diols, the soybean toxins may be at least partly deactivated by subjecting them to high pressure. The process is characterized by the following steps:

- Raw soybeans are comminuted, by either flaking or grinding, to an average particle diameter (or thickness) of 10 mesh or finer;
- The comminuted or ground raw soybeans are intimately admixed with from 0.5 to 5% by



## Poultry Feeds

weight of a diol having from about 3 to 6 carbon atoms; preferred are propylene glycol and butanediol.

(c) The admixture is then compressed under a pressure and for a time sufficient to improve the digestibility of the soybeans (typically found in conventional pelletizing and extrusion equipment).

In combination with the application of pressure, it may be desirable also to provide for the presence of a small amount of moisture. Mild steaming for 10 seconds to 5 minutes with saturated steam is sufficient. In a typical operation, the steamed soybean meal leaving the feed hopper of the pellet mill will be at a temperature of 180° to 185°F., while the temperature of the extruded pellets may range between about 190° and 200°F. The finished material obtained from the foregoing process may either be used in pellet form or be comminuted before blending with the other feed materials.

Example: A series of four soybean-based feeds were prepared of the following composition.

| Ingredients (percent by weight)                               | Diet numbers |       |       |       |
|---|--------------|-------|-------|-------|
|   | 1            | 2     | 3     | 4     |
| Ground yellow corn.....                                       | 51.36        | 51.36 | 51.36 | 51.36 |
| Pelleted ground raw soybeans.....                             | 41.00        |       |       |       |
| Pelleted ground raw soybeans with 1%<br>propylene glycol..... |              | 41.50 |       |       |
| Pelleted ground raw soybeans with 2%<br>propylene glycol..... |              |       | 41.75 |       |
| Pelleted ground raw soybeans with 3%<br>propylene glycol..... |              |       |       | 42.25 |
| Alfalfa meal (20%).....                                       | 2.0          | 2.0   | 2.0   | 2.0   |
| Salt.....   | 0.25         | 0.25  | 0.25  | 0.25  |
| Micronutrient Premix No. 1 <sup>1</sup> .....                 | 0.50         | 0.50  | 0.50  | 0.50  |
| Delamix <sup>2</sup> .....                                    | 0.05         | 0.05  | 0.05  | 0.05  |
| d,l-Methionine.....   | 0.12         | 0.12  | 0.12  | 0.12  |
| Dicalcium phosphate.....                                      | 1.70         | 1.70  | 1.70  | 1.70  |
| Ground limestone.....   | 1.20         | 1.20  | 1.20  | 1.20  |
| Miscellaneous growth factors.....                             | 0.05         | 0.05  | 0.05  | 0.05  |
| Cerelose 2001 <sup>3</sup> .....                              | 1.77         | 1.15  | 0.68  |       |
| Alpha Cel <sup>4</sup> .....                                  |              | 0.12  | 0.34  | 0.52  |

<sup>1</sup> Micronutrient Premix No. 1 supplies the following per pound of finished feed: Vitamin A (U.S.P. units), 2,500; Vitamin D<sub>3</sub> (I.C. units), 500; Vitamin E (Int. units), 1.0; riboflavin (mg.), 2.5; calcium D-pantothenate (mg.), 5.0; choline chloride (mg.), 300.0; Vitamin B<sub>12</sub> (mg.), 5.0; niacin (mg.), 15.0; d,l-methionine (mg.), 227.0; santonin (mg.), 113.5; menadione sodium bisulfite (mg.), 1.0; procaine penicillin (mg.), 2.5; zinc bacitracin (mg.), 2.5.

<sup>2</sup> Delamix provides the following trace elements in the finished feed: Manganese, 60.0 p.p.m.; iron, 20.0 p.p.m.; copper, 2.0 p.p.m.; cobalt, 0.02 p.p.m.; iodine, 1.2 p.p.m.; zinc, 50.0 p.p.m.

<sup>3</sup> Cerelose 2001 is employed as a source of dextrose.

<sup>4</sup> Alpha Cel is a fibrous cellulose employed as a source of inert fibers.

The foregoing formulas are so designed as to provide an isonitrogenous and isocaloric diet. The pelletized ground raw soybeans were processed in a standard pelletizing machine in which the ground raw soybean powder was sprayed with propylene glycol, steamed for about 15 to 30 seconds, and then compressed into pellets. The above described feeds were fed to a series of chicks. The following results were obtained.

| Diet No. | Average gain<br>(grams) <sup>1</sup> | Average feed<br>conversion <sup>2</sup> |
|----------|--------------------------------------|---|
| 1.....   | 649.7                                | 2.21                                    |
| 2.....   | 702.6                                | 2.14                                    |
| 3.....   | 705.7                                | 2.08                                    |
| 4.....   | 703.7                                | 2.11                                    |

<sup>1</sup> Average gain per bird for three replications of ten birds each.

<sup>2</sup> Average of three replications. Each value represents pounds of feed to produce a pound of gain in live weight from day-old to six weeks of age.

## Poultry Feeds

### Chlorohydroxyquinoline

R.B. Edwards (U.S. Patent 2,986,468; May 30, 1961; assigned to Olin Mathieson Chemical Corporation) found that the growth of poultry is substantially accelerated by incorporating a chlorohydroxyquinoline into a conventional poultry feed. The amount of chlorohydroxyquinoline incorporated into the feedstuff may vary from as little as about 4 grams per ton of feed to as much as about 100 grams per ton of feed; the preferred amount being 40 grams per ton of feed.

Example: To illustrate the growth promoting effects of chlorohydroxyquinoline, 160 Broad Breasted Bronze male turkeys were divided into 16 groups, each group consisting of 10 poults. Eight of these groups were fed a high-protein, high-energy basal diet and eight groups were fed the same basal diet containing a mixture of chlorohydroxyquinolines (as naturally obtained by chlorinating 8-hydroxyquinoline) in the ratio of 40 grams of total weight of the chlorohydroxyquinolines per ton of feed. When the poults reached six weeks of age the individual body weights were taken with the results set out below.

| <u>Diet</u>                                       | <u>Final Body Weight, g.</u> | <u>% Increase</u> |
|---|------------------------------|-------------------|
| Basal   | 1,085                        | —                 |
| Basal + 40 g. of CHQ <sup>1</sup> per ton of feed | 1,263                        | 16.41             |

<sup>1</sup> CHQ: A mixture of 5-chloro-8-hydroxyquinoline, and 5,7-dichloro-8-hydroxyquinoline, in proportions resulting naturally from the chlorination of 8-hydroxyquinoline.

### Guanidine

The process of J.H. Hopper (U.S. Patent 3,089,771; May 14, 1963; assigned to Armour and Company) is based on the unexpected discovery that guanidine can be employed as a growth-promotant in poultry feeds. This action of guanidine cannot be explained on the basis of present knowledge. Although guanidine contains amino nitrogens, it is not an amino acid, and poultry, as distinguished from ruminants, are not known to be capable of utilizing non-amino acid nitrogen compounds.

Example: Poultry feed for the process can be prepared by mixing about 70 parts by weight of ground yellow corn with approximately 20 parts by weight of soybean oil meal. There can be also included 3 parts of bone meal, 1 part of limestone, 0.05 part of manganese sulfate, 0.5 part of iodized salt, and 4 parts of tallow. In addition, there should be included about 0.6 part of a standard vitamin premix, which is designed to supply all the vitamins and minerals in excess of the recommended minimum levels. The complete, natural type ration this produced will contain approximately 16% protein. To this feed is then added 0.05% by weight of guanidine hydrochloride, the additive being utilized in the form of a dry powder which is first thoroughly mixed with a small portion of the feed, and the premix thereafter combined and blended with the rest of the feed material. The guanidine-containing poultry feed prepared as just described can then be used in raising of baby chickens for



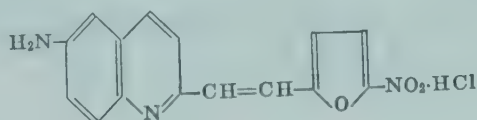
the broiler market. Specifically, the feed can be given to 1 day old chicks on an ad libitum basis, and the feeding continued on this basis for eight weeks. During this period, the feed will comprise substantially the entire diet of the chicks.

### Pyridine Derivatives

P. Schmidt, M. Wilhelm, K. Eichenberger and E. Schumacher (U.S. Patent 3,352,683; November 14, 1967; assigned to Ciba Corporation) found that heterocyclic compounds containing a pyridine ring and in  $\alpha$ -position to the pyridine nitrogen atom, a 5-nitrofuryl-2-methylidene methyl radical, and salts thereof were useful poultry feed additives. Especially valuable are the poultry feedstuffs and feed stuff additives containing 4- or particularly 2-(5'-nitrofurfurylidene-methyl)-pyridines or -quinolines, which may be substituted at the pyridine or quinoline radicals and/or at the ethylene radical or their salts, or N-oxides, and especially those containing 4- or particularly 2-(5'-nitrofurfurylidene-methyl)-quinolines having a free amino group at the quinoline ring. Preferred specific compounds are 2- or 4-(5'-nitrofurfurylidene-methyl)-quinolines, or 2- or 4-(5'-nitrofurfurylidene-methyl)-pyridine, 2-(5'-nitrofurfurylidene-methyl)-6-methyl-pyridine, 4-(5'-nitrofurfurylidene-methyl)-2-amino-quinoline, 2-(5'-nitrofurfurylidene-methyl)-8-amino-quinoline, or particularly 2-(5'-nitrofurfurylidene-methyl)-6-amino-quinoline.

The poultry feedstuffs contain 5 to 100 grams of active substance per ton which makes an average daily dose of about 0.2 to 4 mg. of active substance.

Example 1 — Preparation: A solution of 7.4 g. of 2-methyl-6-amino-quinoline and 6 g. of 5-nitrofurfural in 75 cc of acetic anhydride is heated at 130°C. for 2 hours. The precipitate that forms is filtered off with suction and recrystallized from dimethylformamide + ethanol. There is obtained crystalline 2-(5'-nitrofurfurylidene-methyl)-6-acetamido-quinoline which melts at 284° to 286°C. 12 g. of 2-(5'-nitrofurfurylidene-methyl)-6-acetamido-quinoline are boiled for 3 hours with 120 cc of 2 N-hydrochloric acid in 120 cc of methanol. The precipitate that forms is filtered off with suction, extracted by being boiled with 50 cc of dimethylformamide, and the sparingly soluble 2-(5'-nitrofurfurylidene-methyl)-6-amino-quinoline hydrochloride of the formula



then filtered off with suction. The compound melts above 300°C.

Example 2 — Preparation of poultry feed from the following ingredients:

| <u>Main Ingredients (Premix)</u>                   | <u>Grams</u>           |
|--|------------------------|
| 2-(5'-nitrofurfurylidene-methyl)-6-amino-quinoline | 44.000                 |
| Wheat, medium standard (30-80 mesh)                | 10,956.000             |
| Total Weight                                       | 11,000.000 (continued) |

## Poultry Feeds

| <u>Additives</u>   | <u>Grams</u> |
|--|--------------|
| Cornmeal   | 1,062.875    |
| Fat  | 80.000       |
| Fish meal, 60% protein   | 100.000      |
| Soybean flour, 50% protein   | 500.000      |
| Gluten flour   | 100.000      |
| Alfalfa flour, dried   | 50.000       |
| Corn distillers solubles   | 40.000       |
| Dicalcium phosphate  | 28.000       |
| Calcium carbonate  | 20.000       |
| Iodized salt   | 10.000       |
| Vitamins A and D ( $10^6$ A units and $25 \cdot 10^4$ D units per pound) | 4.000        |
| Calcium pantothenate   | 0.250        |
| Butyl-oxytoluene   | 0.250        |
| Choline chloride of 25% strength   | 2.500        |
| Riboflavin (24 g./pound)   | 0.125        |
| Vitamin B <sub>12</sub> (0.02 g./pound)                                  | 1.000        |
| Methionine   | 0.500        |
| Manganese sulfate  | 0.500        |
| Total weight   | 2,000.000    |

The additives are admixed in the following manner. Approximately half the cornmeal is poured into the mixer, and the remainder is mixed with the heated, liquefied fat and added, and the whole is mixed until the fat has been evenly dispersed. Then manganese sulfate dicalcium phosphate, calcium carbonate and iodized salt are added, and then during the mixing the fish meal, soybean flour, gluten and alfalfa flour and the corn distillers solubles. The batch is thoroughly mixed and then the vitamins A and D, calcium pantothenate, choline chloride, riboflavin, vitamin B<sub>12</sub>, methionine and butyl-oxytoluene are added in this sequence, and the whole is mixed until all the ingredients have been evenly dispersed. When this point is reached, the thoroughly mixed main ingredients are added in an amount such that 50 g. of active substance are evenly distributed in each ton of the feed composition so that average daily intake of active substance is about 2 mg.

### Inorganic Sulfur to Partly Replace Sulfur Amino Acids

The process of R.S. Gordon (U.S. Patent 2,927,859; March 8, 1960; assigned to Monsanto Chemical Company) provides a means of furnishing adequate sulfur in poultry feeds for producing optimum growth response and in which inexpensive inorganic sulfur containing compounds can effectively replace part of the costly sulfur amino acids. These feeds contain at least 75% by weight of the vegetable products and have a protein content of from 20 to 27%.

The proportion of organic sulfur compounds required in the feed composition is that which will provide from 0.20 to 0.30% (based on the weight of the final diet) of sulfur which may be either in the form of naturally occurring sulfur amino acids in the protein, or supplemental



## Poultry Feeds

organic sulfur containing compounds. In the practice of this process poultry feeds which are inherently deficient in sulfur are modified by the addition of sulfur in the form of inorganic compounds to yield a feed containing 0.02 to 0.12% of inorganic sulfur.

Example 1: One month old chicks raised on a commercial 20% protein feed were divided into four groups of equal weight. Two groups were continued on the same commercial feed for four additional weeks, while the other two groups were given the same feed supplemented with 0.45% sodium sulfate. The following table shows the cumulative weight gain (CG); the feed efficiency (FE = wt. feed consumed/wt. gain); and the production efficiency (PE = CG/FE).

|                                       | <u>CG</u> | <u>FE</u> | <u>PE</u> |
|---------------------------------------|-----------|-----------|-----------|
| Control                               | 1,014     | 2.60      | 394       |
| +0.45 Na <sub>2</sub> SO <sub>4</sub> | 1,042     | 2.41      | 433       |

Example 2: Using a feed containing 24.5% protein and 0.58% sulfur amino acids (0.13% S) chicks were grown as described in the preceding examples. The feathering was determined by an arbitrary procedure. The feathering of each fowl was classified in one of four general groups assigned numerical values as follows.

- 1 — Poor
- 2 — Fair
- 3 — Good
- 4 — Perfect

The classifications were made by at least two individuals and the average values for each group of birds is indicated as the Feather score. The following table sets forth the critical data observed.

|   | <u>CG</u> | <u>FE</u> | <u>PE</u> | <u>Feather Score</u> |
|---|-----------|-----------|-----------|----------------------|
| Control   | 318       | 2.09      | 152       | 1.2                  |
| 0.2% Na <sub>2</sub> SO <sub>4</sub> — 0.045% S | 429       | 1.75      | 245       | 2.3                  |
| 0.5% Na <sub>2</sub> SO <sub>4</sub> — 0.11% S  | 425       | 1.75      | 243       | 3.6                  |

### Methionine Substitutes

The aqueous solutions of alkali metal salts of the 2-hydroxy-4-methylthiobutyric acid are useful substitutes for methionine in supplementing low methionine animal feeds, as chemotherapeutic agents for treating dietary ailments and for other purposes usually requiring methionine. R.J. Wineman (U.S. Patent 3,001,874; September 26, 1961; assigned to Monsanto Chemical Company) discusses the use of these methionine substitutes.

Example: To demonstrate the efficiency of the liquid product containing sodium 2-hydroxy-4-methylthio-butyrates as compared to the calcium salt of 2-hydroxy-4-methylthiobutyric acid and methionine, poultry growing experiments were conducted. One day old chicks (Nickols New Hampshire) were sorted by weight and placed 20 per pen in heated batteries.

## Poultry Feeds

At the age of 7 to 10 days, they were weighed again and the 12 chicks closest to average were retained on the feed test. Several groups of the chicks were fed on each of the following rations:

- (1) An experimental feed including isolated soy bean protein, 16% fat, sugar, minerals and vitamins.
- (2) (1) with 0.18% of solid calcium 2-hydroxy-4-methylthiobutyrate prepared by sulfuric acid hydrolysis.
- (3) (1) with dimethionine molecularly equivalent on the acid basis to 0.18% of the calcium salt of (2).
- (4) (1) with the solution of sodium 2-hydroxy-4-methylthiobutyrate in an amount equivalent molecularly on the acid basis to 0.18% of the calcium salt of (2) and prepared by hydrochloric acid hydrolysis.

The chicks were fed for eight weeks and the total gain (in grams) and the feed efficiency (weight of feed consumed weight gain) were measured. The following table shows the averages of the several replications.

| <u>Gain in Grams</u> | <u>Feed Efficiency</u> |
|----------------------|------------------------|
| (2) 452              | 1.61                   |
| (3) 448              | 1.60                   |
| (4) 470              | 1.53                   |

It is apparent that the solution of the sodium salt of 2-hydroxy-4-methylthiobutyric acid is more effective and efficient than the solid calcium salt.

### Better Uptake of Calcium and Phosphate

T.P. Kichline and J.E. Schoolmeester (U.S. Patent 3,121,634; February 18, 1964; assigned to Monsanto Chemical Company) have prepared a formulation which has the advantage that the phosphate therein is readily available to an animal to which it is fed. Feeds formulated therefrom do not interfere with the assimilation of tetracycline antibiotics to the same extent as comparable feeds formulated with dicalcium phosphate as the only source of phosphate material. The composition has the further advantage that it is normally free from caking tendencies and provides a good source of dietary sulfur for poultry.

Example: In suitable mixing equipment there is placed 35 parts of monocalcium phosphate, 45 parts of dicalcium phosphate and 20 parts of calcium sulfate dihydrate. The resulting mixture is thoroughly stirred to provide a concentrate which can be compounded with other animal food ingredients to provide a balanced diet for poultry or other animals. The calcium to phosphorus ratio in this concentrate is 1.25/1; the percent phosphorus is 18.8; the percent calcium is 23.4; and the percent calcium from calcium sulfate is 19.7.

In suitable mixing equipment there is placed 3.5 parts of the above concentrate, 2.5 parts of distillers solubles, 2 parts of dehydrated alfalfa meal, 61 parts of ground yellow corn, 19 parts of soy bean meal, 6 parts of corn gluten meal, 5 parts of fish meal, 0.5 part of sodium chloride, and 0.5 part of a balanced vitamin mixture. A tetracycline antibiotic such



as chlortetracycline or oxytetracycline may be added to the formulation usually at about 200 g./ton of feed. The resulting feed mixture can suitably be fed to poultry of any type and contains calcium and phosphorus in a most desirable ratio for baby turkeys.

### Zinc Nicotinate

M. Hochberg, C.M. Ely and H.C. Klein (U.S. Patent 2,974,043; March 7, 1961; assigned to Nopco Chemical Company) unexpectedly discovered that zinc nicotinate is far more effective than niacin for achieving optimum rate of growth of these animals where niacin has been used before.

Example: Nine lots of chickens, each lot containing sixteen day-old Hubbard New Hampshire cockerel chicks, were fed from one day of age to 28 days of age under a carefully controlled environment, each lot being housed in multideck wire batteries and each lot being provided with the same amount of floor space and the same physical equipment. One of the lots of chicks served as the control lot and was fed a niacin deficient basal diet. It should be noted that the basal diet, although deficient in niacin, contains about 4 mg. per pound of niacin present in the ground yellow corn.

Four lots of the chicks were fed the same niacin deficient basal diet to which was added USP niacin in 5, 10, 20 and 200 mg. quantities per pound of air dried diet respectively. The remaining four lots of chicks were fed the same niacin deficient basal diet to which was added zinc nicotinate in quantities such that the resulting niacin content of the zinc nicotinate was present in 5, 10, 20 and 200 mg. quantities per pound of air dried diet respectively.

The zinc nicotinate which was added had an assay value of 74% by weight of niacin based on the chemical analysis of the zinc nicotinate. All nine lots of chicks were then fed for 28 days under identical conditions. The average gain in weight of the chicks in each lot at the end of the feeding period is given in the following table. The percent response over negative control was calculated from the following:

$$\frac{\text{wt. after 4 wks.} - \text{wt. of control}}{\text{wt. of control}} \times 100$$

| Basal diet plus additive                         | Level of added niacin, mg./lb. | 4 wk. gain, gms. | Percent response over negative control | Percent increased response from zinc nicotinate |
|--|--------------------------------|------------------|--|---|
| Basal diet only (negative control).....          | 0                              | 50.5             |  |   |
| USP Niacin.....                                  | 5                              | 108.7            | 115                                    |   |
| Zinc Nicotinate.....                             | 5                              | 118.8            | 136                                    | +21   |
| USP Niacin.....                                  | 10                             | 128.8            | 155                                    |   |
| Zinc Nicotinate.....                             | 10                             | 130.0            | 158                                    | +3  |
| USP Niacin.....                                  | 20                             | 110.7            | 119                                    |   |
| Zinc Nicotinate.....                             | 20                             | 120.3            | 138                                    | +19   |
| USP Niacin.....                                  | 200                            | 98.9             | 96                                     |   |
| Zinc Nicotinate.....                             | 200                            | 112.0            | 122                                    | +26   |
| Av. increased response from zinc nicotinate..... |                                |                  |  | 17.2  |

Addition of Amino Acids to Low Cost Foodstuffs

C.I. Jarowski (U.S. Patent 3,080,234; March 5, 1963; assigned to Chas. Pfizer & Co., Inc.) shows that a close interrelationship exists between dietary amino acid requirements and the amino acid content of the blood plasma. Although the amino acid content of the plasma is found to change shortly after eating, determination made upon fasting, e.g., about 18 hours after eating, are remarkably reproducible. It has been found that important advantages may be achieved by supplying to an animal a diet whose relative proportions of nutritionally available amino acids conform substantially to the respective proportions of these acids as found in that animal's fasting blood plasma.

Of course, in diet supplementation it may not always be economically practical to bring the relative proportions of all the essential amino acids into complete conformity with the fasting plasma proportions. Important advantages may still be achieved if the essential amino acids present in most limiting proportion in the diet are so adjusted. It will be understood that the first limiting amino acid is that one which is present in the basic diet in the smallest proportion relative to the amino acid profile. The diet is supplemented with respect to the first limiting amino acid to the extent that its total proportion in the dietary protein will balance with the proportion of the second limiting amino acid. When this has been achieved, supplementation with both the first and second limiting amino acids follows, in sufficient quantity to achieve balance with the third limiting amino acid. This process may be continued even further if desired or economically practical.

Example: The levels of essential amino acids in fasting chicken plasma were determined. A corn meal is then supplemented for the feeding of chickens by incorporation therein of 1.13 g. lysine hydrochloride, 0.83 g. threonine and 0.26 g. arginine hydrochloride per 100 g. of meal. The content of available essential amino acids in the feed before and after supplementation is compared with the amino acid profile of the chickens in the table below.

|                  | A<br>Plasma<br>level,<br>mmoles per<br>liter | B<br>Corn meal<br>mmoles per<br>100 g. | C<br>B/A | D<br>Enriched<br>meal,<br>mmoles per<br>100 g. | E<br>D/A |
|------------------|--|--|----------|--|----------|
| Arginine.....    | 0.352  | 2.30                                   | 6.5      | 3.44   | 10       |
| Histidine.....   | 0.126  | 1.48                                   | 12       | 1.45   | 12       |
| Isoleucine.....  | 0.225  | 3.12                                   | 14       | 3.05   | 14       |
| Leucine.....     | 0.253  | 7.85                                   | 31       | 7.68   | 30       |
| Lysine.....      | 0.837  | 2.19                                   | 2.6      | 8.19   | 10       |
| Methionine.....  | 0.093  | 0.80                                   | 10       | 0.78   | 9        |
| Phenylalanine... | 0.119  | 2.30                                   | 19       | 2.25   | 19       |
| Threonine.....   | 0.982  | 2.85                                   | 2.9      | 9.60   | 10       |
| Tryptophan.....  | 0.025  | 0.34                                   | 14       | 0.33   | 13       |
| Valine.....      | 0.231  | 3.50                                   | 15       | 3.42   | 15       |

It is apparent from the table that the available amino acid content of the enriched meal conforms much more closely to the amino acid profile of the chickens than does the corn meal base. Inspection of columns C and E shows that what was a twelve-fold excess of leucine with respect to lysine (31/2.6) has been reduced to only a three-fold excess (30/10).

The supplemented corn meal is a more efficient feed for these chickens than the unsupplemented meal.



Liquid Feed Supplement for Poultry

The liquid feed supplement of R.K. Lindburg (U.S. Patent 3,410,690; November 12, 1968) provides a creamy homogenized emulsion of fats and oils in water together with other materials not readily furnished in solid feed and which increases palatability and metabolism of the oils.

## I

| <u>Syrup</u>                                  | <u>Parts by Weight</u> |
|---|------------------------|
| Emulsifier (Tween 80)                         | 4                      |
| Emulsifier (Neutronyx 600)                    | 4                      |
| Water   | 5                      |
| <u>Feed Liquid</u>                            |                        |
| Syrup   | 1                      |
| Water (1 gal.)                                | 36                     |
| Fat (soya or other vegetable oil)<br>(2 gal.) | 67                     |

## II

| <u>Syrup</u>                                   | <u>Parts by Weight</u> |
|--|------------------------|
| Emulsifier (Tween 80)                          | 4                      |
| Emulsifier (Neutronyx 600)                     | 4                      |
| Emulsifier (C.B. Maraperse)                    | 1/4                    |
| Alcohol  | 2 1/2                  |
| Water  | 5                      |
| <u>Feed Liquid</u>                             |                        |
| Syrup  | 4                      |
| Water (1 gal.)                                 | 36                     |
| Fat (soya or other vegetable oil)<br>(2 gals.) | 67                     |

Tween 80 is a liquid sorbitan monooleate, sp. gr. 1.05-1.10, Neutronyx 600 (also called Osco 500) is a fatty acid ester of a polyglycol, C.B. Maraperse is a lignin-sulfonic compound in the form of a black powder, and all of the above three materials are dispersants and emulsifiers.

The emulsifiers are balanced as to their hydrophilic and lipophilic qualities to produce a stable oil-in-water emulsion and therefore have an HLB of 3 to 6 which, as is well known, is required to make such emulsion. It has been found that addition of one of the lighter edible alcohols reduces both the cost and the effort required in producing a homogeneous emulsifier-water syrup and in emulsifying and homogenizing the final feed. For example, Formula II was used with 2 1/2 parts by weight of ethanol. The weight of the alcohol above stated is a maximum, but any amount up to 2 1/2 parts by weight may be used. Only a small quantity of alcohol is used so that the additional emulsifying effect of the alcohol

be only a matter of solution of the fat, and there is a contraction in volume of the component to which alcohol is added.

Preparation: The syrup is first mixed in a blender to homogenized condition. To the formula amount of syrup is then added the stated amount of water. The fat or oil in liquid condition is then added at the rate of up to two parts per minute while the blender is operating. After all fat has been added the blender is operated for an additional 5 to 10 minutes.

The product is a creamy, yellowish oil-in-water emulsion which is added by a proportioning device to drinking water supplied to animals, at the rate of 16 to 32 oz. per gal. of drinking water. It has been found convenient to supply fats in quantities of more than 20% of the total feed as opposed to the usual 10% maximum which can be properly handled in solid food.

Use: In one series of feeding tests, seven pounds of ordinary solid feed were required to produce a 3 1/2 lb. pullet as compared to a total of 3 lbs. of solid feed and 1 lb. of the present liquid feed for the same weight increase.

### Polyvinylpyrrolidone

V. Dawe (U.S. Patent 3,015,564; January 2, 1962; assigned to Dawe's Laboratories, Inc.) discovered that the rate of growth in the domesticated animals, and especially poultry, can be promoted and accelerated and that the efficiency of utilization of the feed consumed by such means can be increased when a polyvinyl pyrrolidone is added to the diet. PVP is effective when present in the feed in an amount within the range of 0.01 to 0.2% by weight of the feed composition and preferably when present in an amount of 0.05% by weight of the feed. Excellent results have been secured from the use of polymers which are commercially available having a molecular weight average within the range of 40,000 to 360,000.

Example: The following nutritional evaluations were made with turkey poults, all one-day old, broadbreasted bronze poults fed a basal diet.

| <u>Components</u>   | <u>Starter<br/>Composition</u> | <u>Prestarter<br/>Composition</u> |
|---|--------------------------------|-----------------------------------|
| Ground yellow corn  | 920                            | 725                               |
| Dehulled soybean oil meal (50% protein)   | 750                            | 700                               |
| Dried milk with vitamins and antibiotics added<br>(Vitamelk)  | 100                            | 200                               |
| Menhaden fish meal  | --                             | 100                               |
| Meat and bone scraps (50% protein)  | 100                            | 100                               |
| Dehydrated alfalfa leaf meal  | 50                             | 100                               |
| Penicillin feed supplement (4 grams procaine<br>penicillin per pound on calcium carbonate<br>carrier) | --                             | 25                                |
| Dicalcium phosphate   | 50                             | 40                                |
| Salt  | 10                             | 10                                |
| Calcium carbonate   | 20                             | 0                                 |
|   | <hr/> 2,000                    | <hr/> 2,000                       |



## Poultry Feeds

- Group 1 — Group 1 of 12 birds were fed on the starter diet only as a control.
- Group 2 — Group 2 of 12 birds were fed on the starter diet with 0.05% by weight PVP (MW 40,000).
- Group 3 — This group of 12 birds were fed on the prestarter diet only as a control.
- Group 4 — This group of 12 birds were fed on the prestarter diet with 0.05% by weight PVP added.

At the age of four weeks, it was found that the average weight of the 12 birds of group 1 (starter only) was 604 grams whereas the average weight of the 12 birds in group 2 (0.05% PVP) was 626 grams or a gain of 3.6% in weight due to the presence of PVP in the feed. The amount of feed consumed by the birds of group 1 was 1.62 grams per gram of final weight whereas the birds of group 2 consumed only 1.57 grams per gram of final weight. The birds of group 3 had an average weight of 685 grams whereas the birds of group 4 had an average weight of 711 grams, an increase of 3.8% due to the presence of PVP.

It has been further found that PVP is capable also of counteracting some of the undesirable effects of toxic agents incorporated into feeds for various medicinal purposes. For example, the addition of 3-nitro-4-hydroxyphenyl arsonic acid is often incorporated into poultry feed to combat certain diseases. Such arsonic or poisonous compounds impose a stress on poult because of excessive arsenic intake. This stress level is reflected in a marked reduction in the weight gain of the animal and a marked reduction in the rate of weight gain. This and the effect of the combination which includes PVP can be illustrated by the following:

- Group 5 — This group of 12 birds, similar to the birds of groups 1 to 4, were fed on the same starter diet with 3-nitro-4-hydroxyphenyl arsonic acid added in the amount of 0.0198% by weight.
- Group 6 — This group of 12 birds were raised on the feed of group 5 with 0.05% by weight PVP added.

When the birds were four weeks old, the average weight of the birds of group 5 was found to be 446 grams and the average weight of the birds of group 6 was found to be 526 grams. It will be noted that a considerable drop occurred between 604 grams of group 1 and the 446 grams of group 5 which is responsible to the presence of the 3-nitro-4-hydroxyphenyl arsonic acid. It will be observed further that the addition of PVP in the feed of group 6 was effective to increase the weight from 446 grams in group 5 to 526 grams in group 6—a gain of 17.9%. The amount of feed consumed by the birds of group 5 was 1.78 grams per gram of final weight as compared to 1.54 grams per gram of final weight of the birds of group 6.

This is indicative of an increase in the amount of feed consumed in the presence of arsonic acid compound and it is evidence further of a significant decrease when PVP is present, even to a level below that of group 1 without the arsenic.

### Suppressing Gastrointestinal Urease Activity

It has been found that if the activity of urease within the intestinal tract can be suppressed, the growth rate and feeding efficiency of an animal are both markedly increased. W.J. Vissek (U.S. Patent 3,086,864; April 23, 1963; assigned to the U.S. Atomic Energy Commission)

provides a method for suppressing the activity of urease in the intestinal tracts of large numbers of growing animals without the need for making subcutaneous injections into the animals by small repeated dosages over a period of time of certain cyclic acyl substituted ureas having urease suppressant properties. Not all cyclic acyl substituted ureas have suppressant properties, but quite a number of them do, including the class consisting of alloxan, murexide, and barbituric acid. Certain cyclic acyl substituted ureas have hypnotic properties; consequently, a non-hypnotic member having urease suppressant properties should be selected, or at least one whose hypnotic properties are sufficiently weak that no harmful side effects result. The decrease in urease activity is illustrated below.

Example: Four groups of male chicks, each of six chicks except one group of seven, of identical size and breed were housed, fed, and otherwise given the same environmental conditions from birth until sacrifice on their 28th day. The control, or basal group, was given no urease suppressant, and the other three groups had 0.01 mol of suppressant mixed with each kg. of their feed. The suppressant for one group was alloxan, for a second barbituric acid, and for the third murexide. At sacrifice the small intestine was separated from the large intestine and macerated in 0.85% NaCl aqueous solution and then each was placed in a closed vessel and the urease activity was determined by reacting the macerated suspension with a measured amount of urea in a diacidic sodium phosphate buffer. The ammonia thus produced was led to a second vessel containing sulfuric acid. The ammonia in the gases was absorbed by the sulfuric acid and determined as ammonia nitrogen by the standard Kjeldahl procedure. The results of this experiment are set forth below; a unit of "Activity" is defined as the amount of urease required to produce 1 mg. of ammonia nitrogen at 20°C. at neutral pH in a saline phosphate buffer acting upon a urea substrate.

| Diet            | No. of chickens | Small Intestine Activity, Units/gm. |           | Large Intestine Activity, Units/gm. |           |
|-----------------|-----------------|-------------------------------------|-----------|-------------------------------------|-----------|
|                 |                 | Dry Basis                           | Wet Basis | Dry Basis                           | Wet Basis |
| Barb. Acid..... | 1               | 2.88                                | 1.91      | 2.15                                | 7.14      |
|                 | 2               | 2.54                                | 2.02      | 1.74                                | 3.08      |
|                 | 3               | 3.35                                | 1.83      | 2.67                                | 3.24      |
|                 | 4               | 3.41                                | 2.53      | 1.96                                | 3.41      |
|                 | 5               | 2.82                                | 2.44      | 2.99                                | 5.17      |
|                 | 6               | 4.72                                | 3.05      | 3.68                                | 4.50      |
| Average.....    |                 | 3.27                                | 2.30      | 2.53                                | 4.42      |
| Murexide.....   | 7               | 3.02                                | 2.38      | 1.92                                | 5.20      |
|                 | 8               | 2.90                                | 2.70      | 2.49                                | 2.78      |
|                 | 9               | 2.75                                | 2.47      | 2.11                                | 3.84      |
|                 | 10              | 2.53                                | 2.91      | 2.40                                | 5.67      |
|                 | 11              | 3.29                                | 3.39      | 2.50                                | 5.15      |
|                 | 12              | 2.74                                | 1.71      | 2.81                                | 3.26      |
|                 | 13              | 2.78                                | 2.71      | 2.69                                | 6.21      |
| Average.....    |                 | 2.86                                | 2.61      | 2.42                                | 4.59      |
| Basal.....      | 14              | 20.96                               | 16.50     | 3.44                                | 4.89      |
|                 | 15              | 12.95                               | 11.86     | 4.40                                | 4.71      |
|                 | 16              | 46.73                               | 26.62     | 2.76                                | 4.87      |
|                 | 17              | 34.61                               | 29.54     | 2.54                                | 3.93      |
|                 | 18              | 2.94                                | 3.01      | 2.18                                | 4.70      |
|                 | 19              | 3.12                                | 2.03      | 2.48                                | 5.34      |
| Average.....    |                 | 20.22                               | 14.93     | 2.97                                | 4.74      |
| Alloxan.....    | 20              | 3.56                                | 1.85      | 4.04                                | 4.74      |
|                 | 21              | 3.06                                | 2.43      | 2.88                                | 5.65      |
|                 | 22              | 2.75                                | 2.69      | 2.78                                | 6.33      |
|                 | 23              | 2.85                                | 2.61      | 2.61                                | 6.87      |
|                 | 24              | 3.19                                | 2.97      | 2.92                                | 4.97      |
|                 | 25              | 6.29                                | 4.93      | 3.78                                | 7.67      |
| Average.....    |                 | 3.62                                | 2.91      | 3.17                                | 6.00      |



## Poultry Feeds

SUMMARY OF INHIBITION IN CHICKEN GUTS

| Diet                 | No. of Chick-ens | Inhibition in Small Intestine, percent |           | Inhibition in Large Intestine, percent |           |
|----------------------|------------------|--|-----------|--|-----------|
|                      |                  | Dry Basis                              | Wet Basis | Dry Basis                              | Wet Basis |
| Basal (control)..... | 6                |  |           |  |           |
| Alloxan.....         | 7                | 82.09                                  | 80.50     |  |           |
| Barb. Acid.....      | 6                | 83.82                                  | 84.59     | 14.81                                  | 6.78      |
| Murexide.....        | 6                | 85.85                                  | 82.51     | 18.51                                  | 3.16      |

It will be noted from the foregoing table that the percentages of inhibition of urease activity within the small intestine were quite large. No such reduction, of course, is to be found from the data concerning the large intestines, but inasmuch as the wall area of the small intestines is much greater than the wall area of the large intestines, it appears desirable that the suppression of activity take place in the small intestines in order to prevent the diffusion of ammonia through the walls.

### Deodorizing Feed

N. Nakano (U.S. Patent 3,370,953; February 27, 1968) found that the addition of a humic acid or the salt thereof to the feed of livestock and poultry is very effective to deodorize the excrements, to increase the incidence of crossing and breeding, and to promote and beautify the hairs and feathers thereof attractively. The humic acid consists of  $C_5H(4-47)O_{17}$ .

Example: 115 white leghorns of average weight (1,700 g.) were divided into 20 sections, A through T, of which the respective sections measured about 3' by 6' square and all sections were occupied with 5 or 6 leghorns. Throughout a two-month experiment, the following results occurred as indicated in the following tables. Humic acid was given by adding it to the usual feed, in the following amounts:

Groups A to E were fed without the humic acid.

Groups F to J, 0.3% humic acid three times a day.

For groups K to O, 0.2% humic acid was added to the feed and provided three times daily.

For groups P to T, 0.1% humic acid was added to the feed and provided three times a day.

Hens were treated by giving them a feed containing 0.1% of the humic acid. Their quantity of laying-eggs increased from 186 to 322, and a fishy odor was entirely eliminated from the taste of these eggs.

In the table on the following page, Section A represents the first 10 days, Section B represents the second 10 days, Section C represents the third 10 days, and Section D represents the last 30 days.

## Poultry Feeds

|   | Section  | Average weight (g.) | Number of hens and cocks | Volume of the feed eaten per animal (g.) | Quantity of humic-acid added (g.) | Deodorizing effect        | Thickness of the excrements |
|---|----------|---------------------|--------------------------|--|-----------------------------------|---------------------------|-----------------------------|
| A | A-E..... | 1,700-1,900         | 27                       | 100                                      | None                              | Bad odor.....             | Liquid.                     |
|   | F-J..... | 1,700-1,900         | 30                       | 100                                      | 0.3                               | Substantially deodorized. | Substantially thick.        |
|   | K-O..... | 1,700-1,900         | 29                       | 100                                      | 0.2                               | Slight odor.....          | Normal.                     |
|   | P-T..... | 1,700-1,900         | 29                       | 100                                      | 0.1                               | Medium odor...            | Do.                         |
| B | A-E..... | 1,700-1,900         | 27                       | 100                                      | None                              | Bad odor.....             | Liquid.                     |
|   | F-J..... | 1,700-1,900         | 30                       | 100                                      | 0.3                               | Substantially deodorized. | Substantially thick.        |
|   | K-O..... | 1,700-1,900         | 29                       | 100                                      | 0.2                               | Slight odor.....          | Normal.                     |
|   | P-T..... | 1,700-1,900         | 29                       | 100                                      | 0.1                               | Medium odor...            | Do.                         |
| C | A-E..... | 1,700-1,900         | 27                       | 100                                      | None                              | Bad odor.....             | Very soft.                  |
|   | F-J..... | 1,700-1,900         | 30                       | 100                                      | 0.3                               | No smell.....             | Substantially thick.        |
|   | K-O..... | 1,700-1,900         | 29                       | 100                                      | 0.2                               | Medium odor...            | Normal.                     |
|   | P-T..... | 1,700-1,900         | 29                       | 100                                      | 0.1                               | ...do.....                | Do.                         |
| D | A-E..... | 1,700-1,900         | 27                       | 100                                      | None                              | Bad odor.....             | Soft.                       |
|   | F-J..... | 1,700-1,900         | 30                       | 100                                      | 8.0                               | No smell.....             | Thick.                      |
|   | K-O..... | 1,700-1,900         | 29                       | 100                                      | 5.0                               | ...do.....                | Do.                         |
|   | P-T..... | 1,700-1,900         | 29                       | 100                                      | 3.0                               | ...do.....                | Do.                         |

### EGG PRODUCTION

These processes are concerned with obtaining quality eggs in large numbers.

#### Increasing Egg Production Using Hormones in Feed

The object of the process of J.M. Snyder, O.A. Rowoth and C.E. Lee (U.S. Patent 2,945,765; July 19, 1960; assigned to The Beacon Milling Company, Inc.) is to hasten the onset of egg production, and to increase and maintain egg production in poultry by orally administering progesterone as a predeterminedly small part of the diet, viz. in the range of from 0.1 to 25.0 milligrams of progesterone, or an equivalent amount of another compound possessing progesterone activity, per pound of diet. Examples of compounds also useful are desoxycorticosterone, pregnenolone, pregnanediol, dehydroandrosterone, acetoxypregneninolene and 10-nor-progesterone.

Example: Three groups of S.C. White Leghorn pullets, starting at 143 days of age, were fed for 84 days on the following all-mash dietary regimens. Group 1, the control group, received the following all-mash formulation:

|  | <u>Parts by Total Weight</u> |
|--|------------------------------|
| Soybean oil meal (50% protein)                       | 13 1/4                       |
| Alfalfa leaf meal                                    | 2                            |
| Wheat standard middlings                             | 11 1/2                       |
| Wheat red dog  | 2 1/2                        |
| Meat, bone scrap and poultry by-product meal mixture | 2 1/2                        |
| Fish meal  | 1                            |
| Dicalcium phosphate                                  | 1 3/4                        |

(continued)



## Poultry Feeds

### Parts by Total Weight

|  |        |
|--|--------|
| Fortified mixture of dried whey, grain<br>fermentation solubles and dried extracted<br>streptomyces fermentation residue | 2      |
| Salt   | 1/2    |
| Calcium carbonate  | 2 3/4  |
| Beemico  | 1/2    |
| Corn meal  | 44 3/4 |
| Pulverized oats  | 7 1/2  |
| Ground barley  | 5      |
| Animal fat with antioxidants, viz. butylated<br>hydroxyanisole, citric acid and propylene<br>glycol                      | 2 1/2  |

To this was added per ton, 3 lbs. of cod liver oil with added vitamin A and D concentrates, viz. 1,000 units of vitamin A and 600 ICU of vitamin D<sub>3</sub> per gram of cod liver oil. Group 2 received the same diet as group 1 except that 5 milligrams of progesterone was added per pound of feed, and group 3 received the same diet as group 1 except that 10 milligrams of progesterone was added per pound of feed.

During the 84 day experimental period the group 1 birds laid an average of 52 1/4 eggs per bird, the group 2 birds laid an average of 56 1/2 eggs per bird and the group 3 birds laid an average of 57 1/2 eggs per bird.

### Delaying Onset of Egg Laying in Pullets

A pullet egg is one which is too small to command a respectable price in the market, but which costs as much to collect, clean and pack as do the medium and large eggs which command a better price. D.I. Gard and J.E. Wachtstetter (U.S. Patent 3,352,684; November 14, 1967; assigned to Eli Lilly and Company) provide a method for increasing the number of salable eggs produced by hens during their lifetime by administering a progestational agent during the period beginning just prior to the onset of sexual maturity of the hens up to the time when the flock is in 10% egg production between the 126 and 150 day of the life of the pullets in an amount sufficient to delay the onset of egg laying up to 42 days.

Table 1 which follows gives the results of a typical experiment wherein chlormadinone acetate was administered in the feed for twenty-one days to groups of forty H & N pullets at varying times after hatching, and a greater percent of salable eggs was found in the treated groups than in a control group. In the table, column 1 gives the number of grams of chlormadinone per ton of feed, column 2 the date treatment was initiated, column 3 the day treatment was ended, column 4 the average days delay of lay of median hen as compared to corresponding hen for control group, column 5 the average egg mass produced in kg. per hen, and column 6 the percent of large and medium eggs. The eggs upon which the data in columns 5 and 6 are based were collected for about sixty-six weeks after the start of egg production in the control group. The eight grams per ton level of chlormadinone acetate in the feed means that on the average, each pullet received about 0.7 mg. of the progestational agent per day.

## Poultry Feeds

At the 16 g. per ton level in the feed, the daily intake of the progestational agent was about 1.4 mg. per pullet.

TABLE 1:

| Grams/ton    | Treatment        |            | Average Days Delay | Average Egg Mass | Percent Large and Medium |
|--------------|------------------|------------|--------------------|------------------|--------------------------|
|              | Date Initiated   | Date Ended |                    |                  |                          |
| Control..... |                  |            |                    | 17.906           | 92.4                     |
| 8.....       | <sup>1</sup> 126 | 147        | 4.8                | 18.175           | 94.8                     |
| 8.....       | <sup>1</sup> 140 | 161        | 16.6               | 18.275           | 96.5                     |
| 8.....       | <sup>2</sup> 147 | 168        | 21.5               | 17.332           | 94.5                     |
| 12.....      | 147              | 168        | 20.9               | 17.726           | 95.6                     |
| 16.....      | 147              | 168        | 22.8               | 17.925           | 96.1                     |

<sup>1</sup> 2% production.    <sup>2</sup> 10% production.

Table 2 below gives similar data for subcutaneous administration of chlormadinone acetate to groups of forty pullets with eighty pullets in the control group as before. In the table, column 1 gives the treatment level, column 2 the day of injection, column 3 the average days delay of lay of median hen as compared to corresponding hen for control group, column 4 the average egg mass in kg. per hen, and column 5 the percent of large and medium eggs. The eggs upon which the data in columns 4 and 5 are based were collected for about sixty-six weeks after the start of egg production in the control group.

TABLE 2:

| Treatment Level | Day of Injection | Average Days Delay | Average Egg Mass | Percent Large and Medium |
|-----------------|------------------|--------------------|------------------|--------------------------|
| Control.....    |                  |                    | 17.906           | 92.4                     |
| 5 mg.....       | <sup>1</sup> 140 | 9.1                | 17.595           | 94.1                     |
| 5 mg.....       | <sup>2</sup> 147 | 12.1               | 17.968           | 94.2                     |
| 10 mg.....      | 126              | 8.5                | 17.919           | 94.8                     |
| 10 mg.....      | 140              | 13.5               | 18.802           | 93.9                     |
| 10 mg.....      | 147              | 16.8               | 17.594           | 96.0                     |
| 25 mg.....      | 126              | 24.2               | 17.749           | 97.0                     |
| 25 mg.....      | 140              | 26.3               | 17.698           | 96.5                     |
| 25 mg.....      | 147              | 30.0               | 17.177           | 96.4                     |

<sup>1</sup> 2% production.    <sup>2</sup> 10% production.

The control group in the above experiment began to lay eggs at 131 days of age.

In the above tables any increase in percent of large and medium eggs greater than 3% over the control is highly significant statistically.

Intramuscular rather than subcutaneous injection of chlormadinone acetate gives entirely comparable results.



Improved Egg Quality on Addition of Androgens

The most accurate indication of overall egg quality is furnished by albumen height. The albumen in a fresh egg should have a thick gelatinous consistency and stand up around the yolk when the egg is broken out. L.J. Machlin (U.S. Patent 3,438,783; April 15, 1969; assigned to Monsanto Company) found that egg quality as manifested by albumen height is substantially improved by orally administering conventional poultry feed compositions containing androgenic compounds to laying hens.

Example 1: Four groups of sixteen White Leghorn hybrid hens starting at 14 months of age are fed for 84 days on the following feed composition.

| <u>Component</u>                   | <u>Lbs./cwt.</u> |
|------------------------------------|------------------|
| Soybean oil meal (50% protein)     | 18.6             |
| Dehydrated alfalfa meal            | 2.50             |
| Trace mineral mixture <sup>1</sup> | 0.50             |
| Calcium carbonate                  | 5.60             |
| Dicalcium phosphate                | 3.00             |
| Vitamin concentrate <sup>2</sup>   | 1.20             |
| Fat (tallow)                       | 2.00             |
| Methionine hydroxy analog          | 0.05             |
| Ethoxyquin (an antioxidant)        | 0.0125           |
| Yellow corn                        | 66.5375          |

|  |                   |
|--|-------------------|
| <sup>1</sup> The trace minerals consist of:      | <u>Lbs./cwt.</u>  |
| Magnesium sulfate                                | 0.260             |
| Ferric citrate                                   | 0.150             |
| Potassium iodide                                 | 0.005             |
| Zinc chloride                                    | 0.025             |
| Manganese oxide                                  | 0.030             |
| Copper sulfate                                   | 0.030             |
| Total  | <u>0.500</u>      |
| <sup>2</sup> The vitamin supplements consist of: | <u>Mg.</u>        |
| Vitamin A (3,000 USP units/g.)                   | 160,000           |
| Vitamin D <sub>3</sub> (3,000 IC units/g.)       | 18,000            |
| Riboflavin                                       | 312               |
| Niacin   | 2,880             |
| d-Pantothenic acid                               | 1,008             |
| Choline chloride (87% choline)                   | 82,958            |
| Vitamin B <sub>12</sub>                          | 0.72              |
| Menadione sodium bisulfite                       | 43                |
| Thiamine   | 192               |
| Pyridoxine                                       | 312               |
| Folic acid                                       | 60                |
| Biotin   | 9.6               |
| Inert  | 279,505           |
| Total (1.2 lb.)                                  | <u>545,280.32</u> |

## Poultry Feeds

Group 2 received the same diet as Group 1, except that 2.3 milligrams of methyl testosterone are added per pound of feed. Groups 3 and 4 also received the same diet as Group 1 except that 9.1 milligrams and 19.2 milligrams respectively of methyl testosterone are added per pound of feed. All birds are weighed individually at the beginning and end of the experiment. During the fifth and eleventh week of the experiment eggs are collected from all birds on five consecutive days. The eggs are stored overnight at 1°C. On each morning following collection, the eggs are broken out of their shells individually and the albumen height measured with an Ames S-6428 micrometer and Haugh units calculated. Results are given in Table 1 below.

TABLE 1: EFFECT OF METHYL TESTOSTERONE ON EGG QUALITY IN HENS

| <u>Methyl Testosterone<br/>mg./lb.</u> | <u>Haugh Units<br/>(Index of Albumen Height)</u> |
|--|--|
| 0                                      | 72.2   |
| 2.3                                    | 77.5   |
| 9.1                                    | 75.6   |
| 19.2                                   | 76.2   |

Example 2: The procedure of Example 1 is repeated using  $9\alpha$ -fluoro- $11\beta$ -hydroxy- $17\alpha$ -methyl testosterone. Results are given in Table 2 below.

TABLE 2: EFFECT OF  $9\alpha$ -FLUORO- $11\beta$ -HYDROXY- $17\alpha$ -METHYL TESTOSTERONE ON  
EGG QUALITY IN HENS

| <u>Androgenic Material<br/>mg./lb.</u> | <u>Haugh Units<br/>(Index of Albumen Height)</u> |
|--|--|
| 0                                      | 71.7   |
| 0.5                                    | 76.2   |
| 2.5                                    | 78.5   |
| 12.5                                   | 78.8   |

## PIGMENTATION IN POULTRY AND EGGS

The problem of obtaining adequate pigmentation in poultry and eggs has attracted considerable attention in recent years largely because modern methods of raising chickens require the use of high-energy, low-fiber rations which are deficient in the yellow pigments providing the natural color of skin and yolk. Since a well-pigmented broiler is preferred in many areas of the country it is important that the producer be able to supply birds having the desired degree of pigmentation. It is well recognized by poultry nutritionists that the principal pigment involved in pigmenting the skin and body fat of chickens and the yolk of eggs is the carotenoid lutein, generally referred to as xanthophyll (a class term meaning



## Poultry Feeds

hydroxy-carotenoids). The problem of obtaining adequate pigmentation, therefore, is that of finding a suitable source of xanthophyll to include in the rations. To be more specific, the problem is one of finding a source of xanthophyll which is rich in the principal pigmenting compound, lutein.

At the present time the formulation of feeds with adequate pigmenting properties is largely dependent on using feed ingredients which are natural sources of xanthophyll. Yellow corn is the most common source of xanthophyll in poultry feeds; however, the xanthophyll content of this feedstuff is too low to provide optimum pigmentation. Furthermore, in some areas of the country it is desirable to replace the yellow corn with other feedstuffs such as milo or barley which are very low in xanthophyll content. Consequently, it is necessary to add one or more additional sources of xanthophyll to the ration in order to provide optimum pigmentation.

### Algae Meal Rich in Xanthophyll

The process of A.L. Morehouse and R.C. Malzahn (U.S. Patent 3,257,210; June 21, 1966; assigned to Grain Processing Corporation) provides a feed composition comprising a major amount of a nutritive base ration and a minor amount of a dried algae meal sufficient to enhance the skin pigmenting properties of the feed. A preferred composition includes in addition a small amount of a stabilizing agent (antioxidant) which enhances and prevents deterioration of the skin pigmenting properties of the algae meal.

Typical analysis of a preferred algae meal of the culture Spongiococcum excentricum produced under heterotrophic conditions is as follows:

|                  | <u>Percent</u> |
|------------------|----------------|
| Moisture         | 9              |
| Protein          | 32             |
| Fat              | 4              |
| Fiber            | 1              |
| Ash              | 6              |
| <u>Vitamins:</u> | <u>Mg./lb.</u> |
| Riboflavin       | 9              |
| Thiamine         | 12             |
| Beta-carotene    | 250            |
| Niacine          | 35             |
| Choline          | 250            |
| Pantothenic acid | 10             |
| Vitamin E        | 25             |
| Folic acid       | 2              |

Example 1: In order to show the effect of algae on the pigmentation of egg yolks, a dried algae meal was added to a commercial egg ration and fed to caged hens. Each treatment involved four caged hens and after allowing two weeks on the test diets the average yolk color for each hen was determined over a one week period. Yolk color was determined by

measuring the color of an acetone extract on a colorimeter at a wave length of 450 millimicrons. The yolk pigmentation is reported as micrograms of beta-carotene equivalents per gram of tissue. The average results are shown below.

TABLE 1: EFFECT ON YOLK COLOR OF ADDING ALGAE MEAL TO A COMMERCIAL EGG RATION

| <u>Diet</u>        | <u>Pigmentation of Yolk, mcg. Carotene/g.</u> |                |                |
|--------------------|---|----------------|----------------|
|                    | <u>Trial 1</u>                                | <u>Trial 2</u> | <u>Average</u> |
| Basal              | 29.3  | 35.2           | 32.3           |
| Basal + 0.5% Algae | 55.7  | 55.4           | 55.1           |
| Basal + 1.0% Algae | 69.7  | 53.7           | 61.7           |
| Basal + 2.0% Algae | 76.1  | 75.7           | 76.0           |

Example 2: In order to demonstrate the function of the antioxidants in preserving the pigmenting properties of the dried algae meal a poultry feeding test was conducted. The algae-meals employed in this test were stored at room temperature for one year prior to being fed to the chickens.

TABLE 2

| <u>Diet</u>             | <u>Antioxidant</u>                                    | <u>Pigmentation, mcg./g.</u> |
|-------------------------|---|------------------------------|
| Base ration             | None  | 1.4                          |
| Base + 0.75% algae meal | None  | 2.85                         |
| Base + 0.75% algae meal | 0.0075% 1,2-dihydro-6-ethoxy-2,2,4-trimethylquinoline | 7.02                         |

As can be seen from the above results, the feed containing the algae and an antioxidant exhibited excellent pigmenting properties even after storage of the algae for a year.

#### Retinyldene Cyano-Acetic Acid to Enhance Yolk Color

P.H. van Leeuwen (U.S. Patent 3,372,033; March 5, 1968; assigned to North American Philips Company, Inc.) found that a satisfactory improvement in the yolk color can be achieved by using the retinyldene cyano-acetic acid, salt or ester, added to food.

Example 1 — Retinyldene Cyano-Acetic Acid: 70 g. of vitamin A aldehyde (retinene), 30 g. of cyano-acetic acid, 5 g. of ammonium acetate and 5 g. of acetamide in 300 ml. of benzene and 200 ml. of acetic acid were refluxed, while stirring, for four hours and the



## Poultry Feeds

water produced was removed by means of a watertrap. After cooling of the reaction mixture 500 ml. of diethyl ether was added, after which the mixture was washed with water. Then the acid was taken up from the organic liquid by washing it with 500 ml. of an aqueous 1 N NaOH solution. After this water phase had been washed twice with 100 ml. of diethylether, it was acidified with 300 ml. of 2 N sulfuric acid and extracted with 3 x 250 ml. of diethyl-ether. The collected ether extracts were freed from acid by washing, after which the ether was distilled off under reduced pressure. The residue was crystallized from ethanol, so that the crystalline retinyldene cyano-acetic acid having a melting point of 198°C. and a

$E_{1\%}^{1\text{cm}}$  value of 1390 with 442 m $\mu$  was obtained.

Example 2 — Retinyldene Cyano-Acetic Acid Methyleneester: 0.1 mol retinyldene cyano-acetic acid was boiled with 12.5 ml. of dimethylsulfate and 30 g. of K<sub>2</sub>CO<sub>3</sub> in 500 ml. of acetone for one hour while refluxing. The reaction mixture was cooled to about 35°C. and 25 mol concentrated ammonia was added. After being kept for 30 minutes, while shaking occasionally, the reaction mixture was poured out in 1,500 ml. of water and extracted twice with 250 ml. of methylene dichloride. The collected organic layers were washed with 250 ml. of 2 N H<sub>2</sub>SO<sub>4</sub>, 250 ml. of 5% NaHCO<sub>3</sub> solution in water and twice with 250 ml. of water.

The solvent was then distilled off under reduced pressure and the residue was absorbed in 1 liter of boiling methanol. Then the retinyldene cyano-acetic acid methyleneester was caused to crystallize out at room temperature. This ester has an

$E_{1\%}^{1\text{cm}}$  value (453 m $\mu$ ) of 1475 and a melting point of 145° to 145.5°C.

Example 3 — Use: 1 kg. of a poultry food was mixed with 50 g. of finely powdered retinyldene cyano-acetic acid methyleneester. By mixing one part of the food preparation described with 100 parts of the same poultry food without the new coloring substance a poultry food was obtained, which contained the coloring substance in such a concentration that the eggs of hens fed on this new food exhibited a markedly better color of the yolks than those of hens fed on the same food without the coloring substance.

### C<sub>30</sub> Alkyl Ester

A. Gelsendorf and K. Streiff (U.S. Patent 2,940,856; June 14, 1960; assigned to Hoffman-La Roche, Inc.) have found that a feed composition containing as an essential ingredient an alkyl ester of 2,6,11,15-tetramethyl-17-(2,6,6-trimethyl-1-cyclo-hexen-1-yl)-2,4,6,8,10,12,14,16-heptadeca-octaen-1-oic acid in small concentrations will attain the desired yellow hue and intensity in egg yolks and in the skin, shank, beak, fat and flesh of poultry.

Example 1: A basal ration was used. 2,6,11,15-tetramethyl-17-(2,6,6,-trimethyl-1-cyclohexen-1-yl)-2,4,6,8,10,12,14,16-heptadeca-octaen-1-oic acid methyl ester in solution in peanut oil was thoroughly admixed with the basal ration daily in the following proportions:

- (a) 1 mg. C<sub>30</sub> ester (methyl ester) in 10 ml. peanut oil per kg. of basal ration.
- (b) 3 mg. C<sub>30</sub> ester (methyl ester) in 10 ml. peanut oil per kg. of basal ration.
- (c) 6 mg. C<sub>30</sub> ester (methyl ester) in 10 ml. peanut oil per kg. of basal ration.

## Poultry Feeds

Example 2: The feed compositions described in Example 1 containing the different concentrations of C<sub>30</sub> ester were fed according to conventional feeding practice to separate groups of Leghorn laying hens, each group containing 20 hens. The egg yolks from the laying hens were examined for color and compared with the color of the egg yolks obtained from the same hens prior to the use of feed composition containing the C<sub>30</sub> ester. Three days after the use of the feed compositions of Example 1 had begun, a darkening of the egg yolk became manifest. At the expiration of 14 days the final hue was reached and continued from that time. These experiments showed that feed composition (a) resulted in a light yellow coloration of the egg yolks, feed composition (b) effected an intermediate yellow coloring of the egg yolks and feed composition (c) produced a dark yellow coloration of the egg yolks.

Example 3: A group of 10 Leghorn broilers were fed feed composition (c) daily for 82 days beginning immediately after hatching. A control group of 10 similar birds were maintained on the basal ration alone. The two groups of broilers were then killed and compared as to color of skin, shank, beak and flesh. The birds fed composition (c) showed a marked yellow pigmentation which was significantly more intense than in the case of the control group.

### Pyrethrum Plant Wastes as Source of Xanthophyll

H.J. Prebluda, P.F. Wertz and W.H. Hoffman (U.S. Patent 3,333,962; August 1, 1967; assigned to Hoffman-Taff, Inc.) have discovered that the waxy residues obtained from the purification of the pyrethrum extracts provide an economical and acceptable source of xanthophyll, if the waxy residues are first saponified.

Example: 450 g. of a composite waxy residue assaying 3.30 g. xanthophyll and 0.71 g. carotene per pound obtained from pyrethrum crude extract from Ecuador, Kenya and East Africa were hydrolyzed by refluxing for two hours with 400 g. of 50% aqueous KOH and 2 liters of isopropyl alcohol. The resulting reaction mass was cooled and neutralized with 208 cc of concentrated HCl (12 N) to a pH of 7.5. The neutralized mass was further cooled and filtered. The residue on the filter (KCl) was washed with 100 cc of isopropyl alcohol to give a final volume of filtrate of 3.4 liters.

This filtrate analyzed 1.01 milligram of xanthophyll per ml. making a total xanthophyll content of 3.434 g. The 3,400 ml. of filtrate was split into two portions of 1,700 ml. each. One 1,700 ml. fraction was dried in vacuo to provide a residue of 157 g. This was taken up with 500 cc of absolute ethyl alcohol to provide a solution assaying 2.375 milligram of xanthophyll per g. of solution. This solution was then incorporated in a milo basal ration in an amount sufficient to add 35 milligram xanthophyll per pound of feed.

This ration was fed to laying hens and after seven days, there was a significant increase in the egg yolk pigmentation of eggs from these hens. The average NEPA number (obtained by standard procedures developed by National Egg Producers Assn.) of the eggs produced after the first seven days on this ration was 4.25. At the end of 14 days the average NEPA number of the eggs had increased to 6.0 and at the end of the test, after 20 days, the NEPA number of the eggs was 6 1/8.



Xanthophyll Concentrate from Orange Oil

A means of enhancing the xanthophyll content of feed has been developed by N.F. Kruse (U.S. Patent 3,020,159; February 6, 1962; assigned to Central Soya Company, Inc.). A xanthophyll concentrate is obtained from orange oil. The orange oil itself is a by-product of the citrus industry, being usually produced by cold pressing the outer colored rinds of the oranges.

Example 1 — Preparation: Orange oil containing approximately 128 micrograms of xanthophyll per gram of oil was subjected to a single stage extraction with methanol at room temperature (70°F.) and under substantially anhydrous conditions. Two parts by weight of methanol were used per part of oil. The methanol phase containing the extracted xanthophyll was separated from the oil phase. After recovery of the extracted xanthophyll by evaporating the methanol, the xanthophyll concentrate was found to contain approximately 2,860 micrograms of xanthophyll per gram of concentrate.

Example 2 — Use: A typical broiler feed would have the following xanthophyll-supplying ingredients:

|   | <u>Units/lb. of<br/>Finished Feed</u> |
|---|---------------------------------------|
| 70% corn (10,000 units/lb.)               | 7,000                                 |
| 5% corn gluten meal (70,000 units/lb.)    | 3,500                                 |
| 1% dehydrated alfalfa (150,000 units/lb.) | 1,500                                 |
|   | <u>12,000</u>                         |

Replacement of 5% corn gluten meal and 1% alfalfa is made by using 0.4% of the concentrate of Example 1 (2,860 units/gram or approximately 1,300,000 units/lb.). By using 1% of this concentrate in the finished feed, all the necessary xanthophyll can be supplied from this single ingredient. The significance of supplying xanthophyll from a single source would be the feasibility of enabling the manufacturer to formulate feeds with milo by replacing part or all of the corn in the ration. Milo is understood to be the equivalent of corn in feeding value with the exception that it contains practically no xanthophyll.

Xanthophyll from Soybean Soapstocks

N.F. Kruse and W.W. Craven (U.S. Patent 2,924,525; February 9, 1960; assigned to Central Soya Company, Inc.) have discovered that while soybean meal and soybean oil itself has a relatively low xanthophyll content, it is possible to obtain a surprisingly large concentration of stable and easily available xanthophyll in fractions that occur during soybean processing. Certain soybean oil products, such as soapstocks, concentrates obtained by solvent extraction of soapstocks, pigments recovered from decolorization processes of the oil under inert or alkaline conditions, or pigments removed from alkaline bleaching earths and adsorbents, enable an effective feed to be produced of high stability and which can be efficiently utilized by poultry while effecting the desired pigmentation thereof.

In the processing of the soybean oil the following fractions have the indicated xanthophyll content.

## Poultry Feeds

| <u>Ingredient</u>                                     | <u>Xanthophyll, mcg./g.</u> |
|---|-----------------------------|
| Soybean soapstock from crude oil (dry)                | 1,000                       |
| Soybean soapstock from degummed oil<br>(40% moisture) | 1,050                       |
| Soybean soapstock from degummed oil (dry)             | 1,750                       |
| Pigments recovered from alkaline adsorbents           | 3,250                       |
| Pigments from selective solvent decolorization        | 3,700                       |
| Concentrate from soapstock                            | 10,500                      |

The soapstock or the soapstock concentrate has emulsifying action in the intestinal tract which aids better feed adsorption; it eliminates the dustiness of the product, causes the feed to have free-flowing particles, and provides a lubricating effect which reduces frictional heat in pelletizing the feeds. The soapstock has high caloric value, a high unsaturated fatty acid content, gives the feed a better color.

Example: The following diets were fed to chickens and at the end of the feeding test the depot fat of broilers was extracted and analyzed spectrophotometrically at 436 mu.

| Diets   | Xantho-<br>phyll,<br>mcg./gm. | 1% E 1<br>cm. at<br>436 mu. | Pigmen-<br>tation<br>Index <sup>1</sup> |
|---|-------------------------------|-----------------------------|---|
| 1. 70% Wheat Basal.....   | 3.53                          | .0103                       | 63.0                                    |
| 2. 70% Wheat Basal + 2% Degummed<br>Oil Soapstock.....  | 20.85                         | .0331                       | 203                                     |
| 3. 70% Wheat Basal + 4% Degummed<br>Oil Soapstock.....  | 40.00                         | .0473                       | 290                                     |
| 4. 30% Wheat Basal + 40% Corn + 2%<br>Corn Gluten Meal.....   | 16.78                         | .0167                       | 102                                     |
| 5. 30% Wheat Basal + 40% Corn + 2%<br>Corn Gluten Meal + 3% Crude<br>Oil Soapstock.....                         | 27.60                         | .0203                       | 124                                     |
| 6. 30% Wheat Basal + 40% Corn + 2%<br>2% Corn Gluten Meal + 6%<br>Crude Oil Soapstock.....                      | 34.75                         | .0312                       | 191                                     |
| 7. 30% Wheat Basal + 40% Corn + 2%<br>Corn Gluten Meal + 3% Mixed<br>Degummed and Crude Oil Soap-<br>stock..... | 28.00                         | .0261                       | 160                                     |
| 8. 30% Wheat Basal + 40% Corn + 2%<br>Corn Gluten Meal + 6% Mixed<br>Degummed and Crude Oil Soap-<br>stock..... | 38.50                         | .0337                       | 201                                     |
| 9. Control Commercial Broiler Feed..  | 26.33                         | .0163                       | 100                                     |
| 10. 70% Corn Basal + 1½% Degummed<br>Oil Soapstock.....   | 37.10                         | .0258                       | 158                                     |
| 11. 70% Corn Basal + 3% Degummed<br>Oil Soapstock.....  | 50.00                         | .0348                       | 213                                     |
| 12. 70% Corn Basal + 10% Corn Gluten<br>Meal + 3% Alfalfa.....  | 49.25                         | .0284                       | 174                                     |

<sup>1</sup> Percentage values are based on sample #0, a commercial broiler feed at 100.

These results show the direct relationship between xanthophyll content of the diet and pigment deposition in the fowl. It is also apparent that the addition of a soybean xanthophyll source gives a response similar to or better than that obtained by xanthophyll derived from conventional feed ingredients.

### Stable Xanthophyll Feed Composition

C.W. Stewart (U.S. Patent 2,841,495; July 1, 1958; assigned to Corn Products Refining Company) found that xanthophyll which has been recovered from plant sources can be stabilized against oxidative deterioration by absorbing the xanthophyll, dissolved in a



suitable solvent, such as vegetable oil, on deoiled vegetable oil bearing materials, e.g., spent corn or grain sorghum germ meal, and suitably coating this product followed by drying if necessary. The coating may consist of any of the following: steep liquor, and various mixtures of steep liquor and carbohydrates, such as steep liquor-molasses (corn sugar molasses or cane sugar molasses) mixture, steep liquor-dextrose greens mixture, steep liquor-corn syrup mixture, the amount of steep liquor in the mixture being at least about 50%, dry basis. Optionally, there may be used in combination with the aforementioned coating materials for further stabilization a reducing agent or an antioxidant or a combination of the two.

The amount of protective coating, i.e., steep liquor or steep-liquor-carbohydrate mixture, may vary widely, but should be at least about 35%, dry basis, of the entire mixture in order to be effective. From the standpoint of handling the final mixture, it is advisable not to use more than about 60%.

Example — Stabilized Xanthophyll Product on Spent Corn Germ: 360 grams of spent corn germ flakes was warmed to 120° to 140°F. in a steam jacketed mixing bowl. Xanthophyll oil (extracted from corn gluten) was diluted with acidulated soapstock to give a standardized xanthophyll oil containing 1,820 ppm xanthophyll and 706 ppm carotene. An acetone solution containing 1.0 g. DPPD\* was added to 240 grams of the standardized xanthophyll oil and the whole slowly poured into the spent germ with continuous stirring. Complete absorption of the oil by the germ flakes was obtained in about 3 minutes at 120°F. 320 grams of corn sugar molasses (hydrol) (75% DS), 720 grams heavy corn steep liquor (50% DS) and 1.0 grams of sodium bisulfite were added successively to the mix with continuous stirring.

Stirring was continued until the mix was mealy enough to prevent balling in the dryer (about 5 minutes). The product was then dried in a rotary dryer until the outlet temperature had reached about 240°F. (Inlet air temperature was maintained at about 350°F.) The resulting product had a moisture content of 3.5%. Stability data are given below.

### Stability of Xanthophyll at 50°C.

|                                      | Percent Remaining |                |                |
|--------------------------------------|-------------------|----------------|----------------|
|                                      | <u>7 days</u>     | <u>14 days</u> | <u>21 days</u> |
| Initial xanthophyll content, 364 ppm | 99                | 105            | 106            |

\*DPPD = antioxidant N,N-diphenyl-paraphenylenediamine.

## PREVENTION OF MYCOTOXICOSES IN POULTRY

Poultry hemorrhagic syndrome is an example of a mycotoxicoses and is characterized by certain abnormal clinical symptoms and by hemorrhages in the legs, thighs and other organs, as well as by degenerative changes in the liver and kidneys, and by a pronounced depression of the blood-forming tissues. Although death losses as a result of the hemorrhagic syndrome range from 0 to over 50% of the afflicted birds and morbidity up to 90%, the significance of this disease has even greater economic implications. Many chickens which may appear clinically normal are downgraded during dressing, because of hemorrhages in the musculature

of the legs, thighs and breast. J. Forgacs discovered that toxic elaborated products as the result of growth of fungi on cereals fed to animals are the basic cause of this hemorrhagic disease. A principal object of his process is the prevention of those maladies associated with ingestion of cereals, hereinafter defined, such as straw, hay, grain, and processed feed, on which fungi proliferate as saprophytes and give rise to toxic products, either by secretion of metabolic products into the substrata, or by the toxic substance being present as an endotoxin in the mycelium or in the fruiting bodies of the fungus.

Because of the possibility that small portions of infected cereals may stand for a considerable period of time before they are ingested, it is necessary that the cereals be protected against development of toxic fungal metabolic by-products for a long period of time under adverse conditions.

Three processes all developed by J. Forgacs, and assigned to American Cyanamid Company, indicate that the toxic level of these materials could be suppressed. The test procedure is the same for all.

### Use of 2-Bromo-5-Nitrothiazole

This is described in U.S. Patent 3,208,853; September 28, 1965.

Example 1 — Preparation of Animal Feed for Hemorrhagic Syndrome Study: 8 kg. of non-sterile broiler mash were weighed and the moisture content of the feed determined by means of a Delmhorst Moisture Detector. To the sample were added 40 ml. of an aqueous suspension of a mixture of toxic fungi (*Alternaria* sp., *Aspergillus clavatus*, *Aspergillus fumigatus*, *Aspergillus flavus*, *Aspergillus glaucus*, *Penicillium rubrum* and two unidentified species of *Penicillia*) and sufficient tap water free of chlorine to bring the total moisture content of the feed to approximately 21%.

The fungi had been isolated from feed and litter obtained from broiler houses in which the poultry hemorrhagic syndrome was enzootic. The moistened sample was then premixed by hand, rubbed through a 1/8 inch mesh stainless steel screen and mixed mechanically for 5 minutes in a Hobart feed mixer.

The sample was then divided into two lots, and to a small portion of one of the lots a small quantity of 2-bromo-5-nitrothiazole was added. After thoroughly premixing the 2-bromo-5-nitrothiazole with a spatula, more feed was added from the lot from which the small portion was taken, and premixed by hand until the whole lot was premixed, and this was continued until the 2-bromo-5-nitrothiazole content was 300 ppm, based on the weight of the moist feed. The lot containing the 2-bromo-5-nitrothiazole (Lot 2) was then further mixed mechanically for 5 minutes in the Hobart feed mixer. Lot 1, which contained no 2-bromo-5-nitrothiazole, served as a control. Both sample lots were then transferred to round fiber-board containers, equipped with a gauze-covered opening in the lid for ventilation, and incubated at ordinary room temperatures. The sample lots were observed periodically for gross and microscopic proliferation of fungi.

After 14 days, a very heavy fungal growth and formation of fruiting bodies was observed in Lot 1. After 18 days, there was a heavier fungal growth observed and further formation of



fruiting bodies in Lot 1, and no growth was observed in Lot 2. On the 25th day both sample lots were removed and dried for 48 hours in incubators adjusted to approximately 45°C.

Example 2 — Determination of the Efficacy of 2-Bromo-5-Nitrothiazole in the Prevention of Mycotoxicoeses: Two groups of New Hampshire-Red Barred Rock cross, day-old chicks, 20 per group, were fed ad libitum, respectively, the following lots of feed: the inoculum-containing lot with 300 ppm of 2-bromo-5-nitrothiazole, hereinafter called Lot 2, and the inoculum-containing lot, without the 2-bromo-5-nitrothiazole, Lot 1.

On the fourth day of feeding, chicks in the group which were being maintained on the feed of Lot 1, showed signs of the early symptoms of the hemorrhagic syndrome, that is, depression and fetid diarrhea. This condition prevailed until the 17th day when depression was no longer observed; however, diarrhea in this group was observed on the 21st day. Thereafter, chicks in this group grossly appeared normal.

The second group of chicks which were maintained on the feed of Lot 2, in which 2-bromo-5-nitrothiazole was added, showed none of the early symptoms of the hemorrhagic syndrome. The chicks of this group grossly appeared normal for the full period of the test. Additional lots of feed were prepared following the procedure set forth hereinabove. At the end of four weeks, the supply of feed was exhausted, and 10 chicks from each group were sacrificed by decapitation, and examined grossly at autopsy for manifestations of the syndrome.

These test results show that there is suppression of the hemorrhagic syndrome. This is shown by the autopsy, as well as by gross observation which revealed a pronounced suppression of the hemorrhagic syndrome. Attempting to achieve greater lucidity, as well as speed, in the interpretation of the results of the autopsy, an arbitrary "hemorrhagic syndrome" score was assigned to the twelve organs examined at autopsy. A normal organ is given the value zero, slight manifestations 1, moderate manifestations 2, pronounced manifestations 3. The score is then totaled for each of the carcasses examined, and a mean score is determined by dividing the total score of all the carcasses examined by the number of carcasses. The results of the autopsy of the two groups of chicks are set forth in the following table.

|                                       | Lots Tested            |   |
|---------------------------------------|------------------------|---|
|                                       | Lot 1<br>Feed+Inoculum | Lot 2<br>Feed+Inoculum<br>+2-bromo-5-<br>nitrothiazole,<br>300 p.p.m. |
| Number of Chicks Autopsied            | 10                     | 10  |
| Total "Hemorrhagic Syndrome"<br>Score | 160                    | 71  |
| Mean "Hemorrhagic Syndrome"<br>Score  | 16                     | 7   |

Example 3 — Comparative Studies of the Efficacy of Less than Fungicidal Concentrations of 2-Bromo-5-Nitrothiazole and Fungicidal Concentrations of 2-Bromo-5-Nitrothiazole on the Suppression of the Hemorrhagic Syndrome: 9 lots of non-sterile broiler mash containing no antibiotic of coccidiostat were inoculated with a mixture of toxic fungi (*Alternaria* sp., *Aspergillus clavatus*, *Aspergillus fumigatus*, *Aspergillus flavus*, *Aspergillus glaucus*, *Paecilomyces varioti*, *Penicillium purpurogenum*, *Penicillium rubrum*, and three unidentified species of *Penicillia*), adjusted to 21.8% moisture, and containing respectively, 0, 10, 50,

## Poultry Feeds

100, 200, 250, 300, 400 and 500 ppm of 2-bromo-5-nitrothiazole, were incubated at ordinary room temperatures, observed for fungal proliferation over a period of 13 to 14 days, then dried and fed to day-old chicks, 10 animals per group. The chicks, which were maintained on batteries, had free access to water and the prepared feeds, and were observed daily for gross manifestation of toxicity and weighed periodically until the supply of feed became exhausted in any one group. After 4 weeks the feed supply in one group became exhausted. At this time, all chicks in each group were sacrificed by decapitation and examined at autopsy for gross manifestation of poultry hemorrhagic syndrome.

The lots of feed containing from 0 to 100 ppm of 2-bromo-5-nitrothiazole showed profuse fungal proliferation after 14 days. Slight growth was observed in the lots containing 200 to 300 ppm of 2-bromo-5-nitrothiazole after 14 days. No fungal growth was observed in the samples containing 400 and 500 ppm of 2-bromo-5-nitrothiazole. It can be conclusively stated, as a result of autopsy findings, that although the 2-bromo-5-nitrothiazole did not suppress fungal proliferation in the lots containing 10 to 400 ppm of the compound, the chicks that consumed these lots showed a pronounced suppression of the hemorrhagic syndrome.

### Use of 2-Chloro-5-Nitropyridine

This is described in U.S. Patent 3,208,852; September 28, 1965. Examples 1, 2, and 3 were repeated as described above, except that 500 ppm of 2-chloro-5-nitropyridine was used instead of 2-bromo-5-nitrothiazole. The results of the autopsy of the two groups of chicks are set forth in the following table.

|                                    | <u>Lots Tested</u>         |  |
|------------------------------------|----------------------------|--|
|                                    | <u>Lot 1</u>               | <u>Lot 2</u>   |
|                                    | <u>Feed +<br/>Inoculum</u> | <u>Feed + Inoculum +<br/>2-chloro-5-nitro-<br/>pyridine, 500 ppm</u> |
| Number of chicks autopsied         | 10                         | 10   |
| Total "Hemorrhagic Syndrome" Score | 221                        | 22   |
| Mean "Hemorrhagic Syndrome" Score  | 22                         | 2  |

The lots of feed containing from 0 to 100 ppm of 2-chloro-5-nitropyridine showed fungal proliferation after 14 days. No fungal growth was observed in the samples containing 250 and 500 ppm of 2-chloro-5-nitropyridine. It can be conclusively stated, as a result of autopsy findings, that although the 2-chloro-5-nitropyridine did not suppress fungal proliferation in the lots containing 10 to 100 ppm of the compound, the chicks that consumed these lots showed a pronounced suppression of the hemorrhagic syndrome.

### Use of 8-Hydroxyquinoline

This is described in U.S. Patent 3,255,014; June 7, 1966. Examples 1, 2, and 3 were repeated as above, except that 100 ppm 8-hydroxyquinoline was used. The results of the autopsy of the two groups of chicks are set forth in the table on the following page.



## Poultry Feeds

|                                      | Lots Tested                     |  |
|--------------------------------------|---------------------------------|--|
|                                      | Lot 1,<br>Feed Plus<br>Inoculum | Lot 2,<br>Feed Plus<br>Inoculum<br>Plus 8-hy-<br>droxyquino-<br>line, 100 p.p.m. |
| Number of Chicks Autopsied.....      | 10                              | 10   |
| Total "Hemorrhagic Syndrome" Score.. | 160                             | 71   |
| Mean "Hemorrhagic Syndrome" Score..  | 16                              | 7  |

The lots of feed containing from 0 to 100 ppm of 8-hydroxyquinoline showed profuse fungal proliferation after 14 days. Slight growth was observed in the lots containing 200 to 300 ppm of 8-hydroxyquinoline after 14 days. No fungal growth was observed in the samples containing 400 and 500 ppm of 8-hydroxyquinoline. It can be conclusively stated, as a result of autopsy findings, that although the 8-hydroxyquinoline did not suppress fungal proliferation in the lots containing 10 to 400 ppm of the compound, the chicks that consumed these lots showed a pronounced suppression of the hemorrhagic syndrome.

### VITAMIN K ACTIVE MATERIALS

Vitamin K is in general use in many animal feeds, particularly in poultry feed, where its effect is to eliminate hemorrhagic disease in the bird. These processes are concerned with making vitamin K active preparations which retain their stability over a long period of time. In these processes MBA refers to menadione bisulfite adduct; MSB refers to menadione sodium bisulfite.

#### Vitamin K Premix on a Cornmeal Carrier

The premix of this process is prepared by simply forming a dry mixture of menadione bisulfite adduct, preferably menadione sodium bisulfite, and corn meal in the desired proportions. For example, 16 g. of MSB are added to sufficient corn meal to make a pound of the premix. By this simple procedure, premixes can be prepared containing from a minimum of about 4 g. to a practical maximum of about 250 to 260 g. MBA per pound. (R. Berruti; U.S. Patent 3,079,261; February 26, 1963; assigned to Heterochemical Corporation).

Example 1: In order to compare stability or vitamin K potency retention, typical premixes of average concentration (16 g. MSB per pound), were prepared employing three widely used carriers. The potency retention figures are reported in the following table.

TABLE 1: CARRIERS

| [Percent vitamin K potency retention]             |              |                  |                        |         |
|---|--------------|------------------|------------------------|---------|
| Aging Conditions (Time at<br>95° F. and 95% R.H.) | Corn<br>Meal | Soy bean<br>Meal | Wheat<br>Mid-<br>lings | Calcite |
| 60 hours.....                                     | 80.9         | 60.3             | 68.2                   | 33.2    |
| 90 hours.....                                     | 74.6         | 39.2             | 47.5                   | 27.3    |
| 120 hours.....                                    | 70.8         | 36.4             | 42.5                   | 27.2    |

## Poultry Feeds

The aging conditions employed were no more drastic than the premix would be subjected to upon standing in a very hot, moist climate.

Example 2: In this series of tests the samples were subjected to accelerated aging. Specifically, the samples were steamed for one and one-half minutes at seven and one-half pounds steam pressure. This steaming is equivalent to normal aging in air for a period of sixty hours at 115°F. and 80% RH. The results of these tests were as follows.

**TABLE 2: CARRIERS**

| [Percent vitamin K potency retention] |           |               |                 |
|---------------------------------------|-----------|---------------|-----------------|
| MSB Premix (Approx. g./lb.)           | Corn Meal | Soy bean Meal | Wheat Middlings |
| 4.....                                | 64.7      | 47.1          | 54.5            |
| 8.....                                | 60.0      | 39.8          | 51.6            |
| 16.....                               | 75.1      | 42.1          | 66.7            |
| 32.....                               | 76.6      | 62.0          | 70.7            |
| 64.....                               | 79.2      | 58.9          | 72.6            |

From the above it can be seen that at all dilutions the MSB-corn meal premix exhibits improved stability or vitamin K potency retention. While the preferred premix employs a carrier which is all corn meal, it has been found that the carrier may be a mixture of corn meal and wheat middlings and/or soy bean meal, with corn meal constituting at least 50% by weight of the total carrier.

### Monocalcium Phosphate as Stabilizer

W. Galler (U.S. Patent 3,079,260; February 26, 1963) found that the initial vitamin K potency of the premix can be stabilized to a great extent and deterioration with time and in the presence of moisture and air, i.e., relatively high humidity, can be greatly inhibited by incorporating in the premix an acid reacting material which produces a more acidic water extract. A preferred stabilizer is monocalcium phosphate.

Example 1: The following table shows the effect of the addition of 4, 8, 12, and 24 grams of monocalcium phosphate monohydrate to a typical 4 gram MSB calcite premix after storage for approximately three months at normal conditions of temperature and relative humidity.

**TABLE 1: CALCITE PREMIX —  $\text{CaH}_4(\text{PO}_4)_2$  STABILIZER**

| Ex.    | MSB, g./lb. | Stabilizer, g./lb. | Original assay |      | Aging time, weeks | Aged assay |     | Vitamin K potency, percent retention |
|--------|-------------|--------------------|----------------|------|-------------------|------------|-----|--------------------------------------|
|        |             |                    | Percent M      | pH   |                   | Percent M  | pH  |                                      |
| 1..... | 4.17        | -----              | 0.29           | 7.5  | 13                | 0.18       | 8.2 | 62                                   |
| 2..... | 4.75        | 4                  | 0.33           | 5.9  | 13                | 0.21       | 6.3 | 64                                   |
| 3..... | 4.03        | 8                  | 0.28           | 5.75 | 13                | 0.19       | 5.9 | 64                                   |
| 4..... | 4.32        | 12                 | 0.30           | 5.4  | 13                | 0.22       | 5.6 | 73                                   |
| 5..... | 3.75        | 24                 | 0.26           | 4.7  | 13                | 0.26       | 5.0 | 100                                  |
| 6..... | 3.75        | 24                 | 0.26           | 4.8  | 12                | 0.26       | 5.1 | 100                                  |



Example 2: The following table shows the potency of premixes containing 16, 32 and 64 g. per pound MSB, respectively.

TABLE 2: CALCITE CARRIER

| Ex.   | MSB,<br>g./lb. | Stabilizer<br>$\text{CaH}_2(\text{PO}_4)_2$ ,<br>g./lb. | Original<br>assay |     | Aging<br>time,<br>weeks | Aged<br>assay     |     | Vitamin<br>K<br>potency,<br>percent<br>retention |
|-------|----------------|---|-------------------|-----|-------------------------|-------------------|-----|--|
|       |                |   | Per-<br>cent<br>M | pH  |                         | Per-<br>cent<br>M | pH  |  |
| 7---- | 16             | 4   | 1.03              | 6.3 | 8                       | 0.85              | 7.0 | 82   |
| 8---- | 32             | 4   | 2.14              | 6.6 | 8                       | 1.97              | 7.3 | 92   |
| 9---- | 64             | 12  | 4.25              | 5.5 | 8                       | 4.14              | 6.9 | 97   |

It is thus seen that the more dilute the premix with respect to MSB, the more monocalcium phosphate stabilizer is required to effect substantially complete protection of the vitamin K active material, and this quantity generally will not exceed about 24 grams per pound with a calcite carrier. However, with a concentrated premix only 4 grams are required. At accelerated aging conditions monocalcium phosphate substantially improves the stability of the vitamin K active material of a MSB wheat midlings premix. Sodium acid pyrophosphate and potassium pyrosulfate are on a par with monocalcium phosphate as stabilizers. Terephthalic acid when added to monocalcium phosphate showed stabilizing properties.

#### Coated Menadione Bisulfite Adduct

In another process, W. Galler (U.S. Patent 3,196,018; July 20, 1965) provides a coating or protective film for the finely divided MBA particles which is substantially impermeable to moisture, thus protecting the MBA. With a moisture barrier erected between the MBA and its surroundings, for example, the other ingredients of the feed which may supply the alkali and/or moisture necessary for decomposition, the moisture of the air or the moisture content of the other feed ingredients carrying small quantities of minerals and alkali cannot reach the MBA.

The coating or film material that surrounds the particles of the water-soluble menadione bisulfite adduct is non-toxic and substantially impermeable to moisture, yet it is readily penetrated by digestive fluids present in the stomach of the animal. Several film materials have been found to produce excellent results, among which are methyl cellulose, ethyl cellulose, edible shellac, waxes, for example, glyceryl monostearate, other polyhydric fatty acid esters, hydrogenated tallow, cellulose acetate phthalate and polyvinyl fatty acid esters such as polyvinyl stearate. Two preferred materials are ethyl cellulose and shellac.

As a general rule, at least 4 grams stabilizer will be employed per pound of premix, although stabilizing activity has been observed with the more strongly acidic stabilizers when present in amounts less than 4 grams per lb. A range of about 12 to 36 grams will adequately protect the very dilute as well as the concentrated premixes.

Example 1: To 1,000 g. of an ethanol solution of edible shellac, i.e. pharmaceutical glaze,

containing four pounds of shellac per gallon, there was added 1,110 g. of MSB and 1,000 cc of isopropyl alcohol. The alcohol was added to thin the solution sufficiently to enable spray drying. The dried product assayed 87.5% MSB and upon compounding into a feed and pelletizing the pellets showed a loss of vitamin K potency after 7 days of only 6.7%.

Example 2: Five parts of MSB and one part of monocalcium phosphate, a stabilizer for MSB, were dissolved and suspended in an aqueous phase containing 3% methyl cellulose. This suspension was then spray dried to produce a particle in which MSB and monocalcium phosphate were bound together by the methyl cellulose. This material can be used as is in feed, but better results are obtained by proceeding further, as follows: The dried material was then slurried in a mixture containing 400 parts isopropyl alcohol, 400 parts of Hyarofol Glyceride T-57 (Archer-Daniels-Midland Co. brand of hydrogenated tallow containing about 70% C18), 40 parts of soybean flour, 8 parts of a wetting agent, 5,360 parts of methylene chloride as a solvent for the hydrogenated tallow. The resulting slurry was then spray dried to produce an MSB particle having an inner core of MSB and monocalcium phosphate and a fatty outer coating or film of hydrogenated tallow in which there were dispersed particles of soybean flour and the surface active agent.

As the product is ingested, the soybean meal will very rapidly be digested, leaving pores in the particle so that the digestive juices will quickly break up the remaining particle and render the MSB readily available to the animal. The resulting spray dried complex product contained 84 1/2% MSB. Upon compounding into a feed and pelletizing and after the passage of 7 days, the vitamin K activity of the pellets was reduced by only about 10.4%.

### Menadione Bisulfite Adducts of Organic Nitrogenous Bases

Menadione derivatives for use in Vitamin K active compositions such as chicken feeds should preferably have the following properties: (1) they should not add water of crystallization, (2) they should be only slightly soluble in water and (3) their saturated aqueous solutions should have a hydrogen ion concentration conducive to a low rate of decomposition, preferably corresponding to a pH value of less than 4.5. Such derivatives can be obtained by reaction between menadione sodium bisulfite or other water soluble bisulfite adducts and certain weakly basic organic compounds.

The adducts prepared in the process of J.B. Nanninga (U.S. Patent 3,328,169; June 27, 1967; assigned to Heterochemical Corporation) meet these requirements. The preferred nitrogenous organic bases are triazine, pyrimidine and guanidine derivatives.

Example 1: A solution of 5 grams of the sulfate salt of dicyanodiamidine sulfate in 100 ml. of water is mixed with a solution of 9.3 grams of menadione sodium bisulfite in 15 ml. of water. Almost at once the menadione adduct of dicyano-diamidine bisulfite compound crystallizes out; after standing for a while the crystals are filtered by suction, washed with water and dried at a temperature of 60°C. The yield is 8.5 grams of white crystals, sparingly soluble in water. The saturated aqueous solution shows a pH less than 4.5 and their moisture content is less than 1%.

Example 2: A lukewarm solution of 2.5 grams of 2,4,6-triamino-1,3,5-triazine in 110 ml. of 0.4 N hydrochloric acid is mixed with a solution of 9 grams of menadione sodium bisulfite



## Poultry Feeds

in 20 ml. of water. A thick precipitate of the menadione bisulfite adduct of 2,4,6-tri-amino-1,3,5-triazine is immediately formed. After standing for some time, it is filtered by suction, washed with water and dried at a temperature of 60°C. There is obtained 7.5 g. of white crystals, almost insoluble in water. Their moisture content is less than 1% and the saturated aqueous solution shows a pH less than 4.5.

Example 3: A solution of 1.26 grams of 2,4-diamino-6-hydroxy-pyrimidine in 15 ml. of 1.3 N HCl is mixed with a solution of 3.5 grams of menadione sodium sulfite in 10 ml of 0.5 N HCl. Almost immediately a precipitate is formed of the menadione 2,4-diamino-6-hydroxy-pyrimidine bisulfite compound. The latter is filtered by suction, washed with water and dried at a temperature of 60°C. The yield is 3.5 grams of white crystals, sparingly soluble in water. Their moisture content is less than 1% and their saturated aqueous solution has a pH lower than 4.5.

## DUCK FEEDS

The trend in modern human nutrition is toward the consumption of diets containing reduced amounts of animal fats. In line with this trend, animal nutritionists have sought ways to make the domesticated duck less greasy when cooked and therefore more acceptable to the modern palate. In the three processes which follow, the basal feed ration was the same and contained the following:

|                                   |       |               |
|-----------------------------------|-------|---------------|
| Ground yellow corn                | 1,468 | lbs.          |
| Ground oats                       | 60    | lbs.          |
| Soybean oil meal, 44%             | 99    | lbs.          |
| Fish meal, 60%                    | 150   | lbs.          |
| Dried fish solubles               | 10    | lbs.          |
| Dried whey                        | 40    | lbs.          |
| B·Y Basic <sup>1</sup>            | 40    | lbs.          |
| Alfalfa meal, 17%                 | 50    | lbs.          |
| Dicalcium phosphate               | 30    | lbs.          |
| Ground limestone                  | 40    | lbs.          |
| MnSO <sub>4</sub> , feed grade    | 0.5   | lbs.          |
| CCC trace mineral <sup>2</sup>    | 0.5   | lbs.          |
| Iodized salt                      | 5     | lbs.          |
| Vitamin A (10,000 u.)             | 272   | g.            |
| Vitamin D <sub>3</sub> (1,500 u.) | 4     | lbs.          |
| B·Y-21 <sup>3</sup>               | 1     | lbs.          |
| Alpha-tocopherol acetate          | 7     | g.            |
| Niacin                            | 40    | g.            |
| Menadione                         | 0.5   | g.            |
| Proferm-6 <sup>4</sup>            | 1     | lbs.          |
| Baciferm PB-10 <sup>5</sup>       | 1     | lbs.          |
| <u>Calculated Analysis</u>        |       |               |
| Protein                           | 15.0  | %             |
| Fat                               | 3.6   | % (continued) |

## Poultry Feeds

|                        |       |           |
|------------------------|-------|-----------|
| Fiber                  | 3.4   | %         |
| Calcium                | 1.63  | %         |
| Phosphorus             | 0.77  | %         |
| Energy                 | 1,000 | cal./lb.  |
| Riboflavin             | 3.29  | mg./lb.   |
| Niacin                 | 32.1  | mg./lb.   |
| Pantothenic acid       | 5.3   | mg./lb.   |
| Choline                | 363   | mg./lb.   |
| Vitamin A              | 4,594 | units/lb. |
| Vitamin D <sub>3</sub> | 1,361 | units/lb. |

1 Commercial Solvents Corporation commercial fermentation residues containing unknown growth factors.

2 Calcium Carbonate Company trace mineral salt having a guaranteed analysis of:

|                    |        |
|--------------------|--------|
| Manganese, minimum | 12.20% |
| Iron, minimum      | 9.60%  |
| Calcium, maximum   | 9.50%  |
| Calcium, minimum   | 7.50%  |
| Copper, minimum    | 0.73%  |
| Zinc, minimum      | 0.67%  |
| Iodine, minimum    | 0.38%  |
| Cobalt, minimum    | 0.26%  |

3 B-Y-21 is Commercial Solvents Corporation riboflavin feed supplement containing 8 mg. of riboflavin per gram of supplement.

4 Proform-6 is Commercial Solvents Corporation feed supplement containing 6 mg. of vitamin B<sub>12</sub> per pound of supplement.

5 Baciferm PB-10 is Commercial Solvents Corporation feed supplement additive containing 7.5 grams of bacitracin per pound and 2.5 grams of procaine penicillin per pound.

### Effect of Nitroalcohols and Nitrocarbamates to Reduce Fat

M.C. Bachman, J.L. Martin and J.M. Pensack (U.S. Patent 2,924,526; February 9, 1960; assigned to Commercial Solvents Corporation) developed a feed containing certain nitroalcohols and carbamates.

Example 1: Duplicate lots of 15 day oil Pekin ducks were fed basal ration. At the end of 43 days the ducks were weighed, sacrificed, defeathered, finely ground and homogenized to form a liquid blend which was then ether extracted and analyzed. The ducks weighed on an average of 6.18 pounds at the end of the test and had consumed 2.95 pounds of feed per pound of weight gain. The duck carcasses had the following composition: 35.2% fat, 12.8% protein, 50.2% water, and 2.7% ash.



## Poultry Feeds

Example 2: The procedure of Example 1 was carried out except that 100 grams of 2-nitro-2-methyl-1-propanol were added to the basal ration by blending the alcohol into the feed during mixing. At the end of the 43 day test period the ducks averaged 6.25 pounds of weight and had consumed 2.94 pounds of food for every pound of weight gain. The duck carcasses had the following composition: 32% fat, 12.1% protein, 53.0% water and 2.9% ash.

Example 3: The process of Example 1 was followed except that 100 grams of 2-nitro-2-propyl-1,3-propanediol dicarbamate were added to the ration of Example 1. At the end of the 43-day test period the ducks averaged 5.88 pounds weight and had gained one pound of weight for every 2.98 pounds of feed consumed. The ducks were found to have the following body compositions: 32.7% fat, 12.8% protein, 52.2% water, and 2.5% ash.

### Effect of Dinitroalcohols and Carbamates to Reduce Fat

This procedure by J.L. Martin (U.S. Patent 2,924,527; February 9, 1960; assigned to Commercial Solvents Corporation) is very similar to the one discussed above.

Example 4: Duplicate lots of 15 day old Pekin ducks were fed basal ration as described in Example 1 above. Meanwhile, another duplicate lot of 15 ducks are fed the same feed to which 100 g. of 2,2-dinitropropanol has been added. At the end of 43 days the ducks are weighed, sacrificed, defeathered, finely ground, homogenized to form a blend, and the fat ether extracted therefrom. In each instance the additive-containing feeds give a 5 to 10% by weight decrease in the amount of total fat when ducks fed the additive-supplemented feeds are compared with ducks fed the described ration containing no additives.

### Propionic Acid as Growth Stimulator

P.D. Bogdonoff, G.W. Thrasher and J.N. Henson (U.S. Patent 3,219,453; November 23, 1965; assigned to Commercial Solvents Corporation) fed control ducks basal ration as in Example 1 above. At the end of 41 days, the ducks were weighed and sacrificed. The ducks had gained an average of 7.53 pounds at the end of the test and had consumed 2.98 pounds of feed per pound of weight gain.

Example 5: The procedure of Example 1 was carried out except that 4.5 kilograms of propionic acid were added to the basal ration. At the end of the 41-day test period, the ducks had gained on the average of 7.93 pounds and had consumed 2.62 pounds of food for every pound of weight gained.

Example 6: The procedure of Example 1 was carried out except that 9.0 kilograms of propionic acid were added to the basal ration. At the end of the 41-day test period, the ducks had gained on an average of 7.93 pounds and had consumed 2.62 pound of food for every pound of weight gained.

## RUMINANT FEEDS

### STIMULATING RUMEN ACTIVITY

It has been known that the chemical compositions of the rumen fluids will reflect the well being of the ruminant and will provide a means of ascertaining the feed efficiency, the weight gain of the animal, and the adequacy of the feed. Rumen fluids contain a substantial proportion of volatile fatty acids, including acetic, propionic and butyric acids. These acids are derived from carbohydrate fermentation by rumen microflora and provide the principal source of energy to the animal. Of these fatty acids, the propionic acid content is most indicative of feed efficiency and weight gain. Increases in this component will be reflected immediately by improved feed utilization by the ruminant. The rumen microflora (yeasts, protozoa and bacteria) are individually capable of converting vegetable protein to amino acid and of converting nonprotein nitrogen into proteins. The microflora are also capable of digesting cellulose feeds.

### Olefins

The process of E.S. Erwin and G.J. Marco (U.S. Patent 3,328,168; June 27, 1967; assigned to Monsanto Company) is concerned with the addition of olefins to ruminant feeds. Using in vitro fermentation reaction studies in which conditions existing in the rumen are simulated, and propionate content measured by chemical analysis it was found that olefins added to ruminant feeds will stimulate growth and result in a more efficient utilization of feed components that will occur when the ruminants are fed with the identical feed without the olefins. The useful olefins are the unsaturated hydrocarbons with either branched or straight hydrocarbon chains of 6 to 20 carbon atoms. Preferred types are the straight chains with terminal unsaturation, the branched olefins with methyl side chains and the polymer of olefins having from 3 to 6 carbon atoms, such as propylene, butylene, pentenes and hexenes. It has been found that from 0.1% by weight to 2.0% will produce a stimulation of rumen microflora and increase in propionate.

Example 1: A series of hydrocarbon olefins were studied to determine their effect in vitro in increasing the propionic acid content of rumen fluids by such in vitro procedure. Controls were run by identical procedures wherein the olefins were not included. The observed differences in mol percent increase due to the presence of the olefin is set forth in the following table.



## Ruminant Feeds

| Olefin  | Propionate response<br>increase in percent of control |
|---|---|
| Propylene dimer .....                                   | 5.1   |
| 2,5-dimethyl hexadiene .....                            | 8.1   |
| 1-tetradecene .....                                     | 7.1   |
| Diisobutylene .....                                     | 1.1   |
| Hexadecene .....  | 5.8   |
| C <sub>10</sub> to C <sub>18</sub> olefin mixture ..... | 6.4   |
| n-Hexene dimer .....                                    | 2.8   |
| Propylene trimer .....                                  | 4.4   |
| 1-hexene-1-heptene codimer .....                        | 2.0   |
| 1-ercosine .....  | 3.3   |

**Example 2:** A variety of olefins was studied in vivo to determine the percent increase in propionate when added to a normal diet. The rumen fluid analysis was made on fluids withdrawn from sheep fed a standard diet. The diet was then changed by including 2% by weight of an olefin and 20 hours later the rumen fluid was withdrawn and analyzed. The percent of increase was calculated and recorded in the following table.

| Olefin                                 | Percent<br>in Diet | Increase<br>Percent<br>Propionic<br>Acid |
|--|--------------------|--|
| Triisobutylene .....                   | 2.0                | 158                                      |
| 1-dodecene .....                       | 2.0                | 148                                      |
| n-Hexene dimer .....                   | 2.0                | 130                                      |
| 2-methylpentene dimer .....            | 2.0                | 149                                      |
| Propylene trimer .....                 | 2.0                | 144                                      |
| Propylene tetramer .....               | 2.0                | 170                                      |
| Propylene pentamer .....               | 2.0                | 135                                      |
| Tetrapropenyl succinic anhydride ..... | 1.0                | 148                                      |

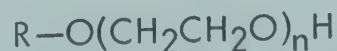
**Example 3:** Sheep were fed a normal diet containing 1% of 1-dodecene or 1% of propylene tetramer. After a predetermined period of time the sheep were weighed and compared with a control group of sheep fed the same diet except without the olefins. The following observations of feed per unit of weight increase were noted.

|                          | Feed/gain in wt. |
|--------------------------|------------------|
| Control .....            | 10.86            |
| 1-dodecene .....         | 8.87             |
| Propylene tetramer ..... | 9.78             |

A suitable ruminant feed to which the preferred olefins are added could contain cellulosic roughage, natural oils, antioxidants, minerals, vitamins and medicants.

### Ethoxylated Alcohols

In a later process E.S. Erwin and G.J. Marco, (U.S. Patent 3,421,898; January 14, 1969; assigned to Monsanto Company) found that the growth of ruminants is stimulated by feeding the ruminants a feed composition containing an ethoxylated alcohol of the formula;



wherein R is alkyl of at least 9 and not more than 20 carbon atoms and n is an integer of at least 2 and not more than 20.

## Ruminant Feeds

Example 1: Again in vitro fermentation reactions used in measuring the effect of feed additives on normal ruminant diets a wide variety of alcohols condensed with ethylene oxide were studied to determine the increase of propionic acid induced by the rumen microflora in the presence of the ethoxylated alcohols. The results of this study are shown in the following table.

| Alcohol   | Mols of EO | Propionate |
|---|------------|------------|
| n-Octyl.....  | 5.2        | -0.6       |
| Do.....   | 15.0       | -0.6       |
| n-Decyl.....  | 7.5        | 10.6       |
| Do.....   | 29.7       | -1.3       |
| Normal alcohols having an average of 10 carbon atoms.....       | 4          | 11.8       |
| Dodecyl alcohol.....  | 1          | 0.7        |
| Do.....   | 3          | 7.2        |
| Do.....   | 7.5        | 15.4       |
| Do.....   | 25         | -0.6       |
| Mixed normal alcohols having an average of 12 carbon atoms..... | 4          | 15.5       |
| n-Tetradecyl alcohols.....                                      | 8.1        | 12         |
| Do.....   | 15         | 14         |
| Do.....   | 29.8       | -1.9       |
| Mixed normal alcohols with an average of 14 carbon atoms.....   | 4          | 10.4       |
| n-Hexadecyl.....  | 4          | 4.5        |
| Do.....   | 20         | 7.3        |
| Octadecyl.....  | 14.7       | 4.7        |
| Mixed normal alcohols with an average of 18 carbon atoms.....   | 4          | 2.5        |
| Mixed normal alcohols having 12 to 15 carbon atoms.....         | 4          | 12.6       |
| Mixed normal alcohols having 11 to 16 carbon atoms.....         | 4          | 12.6       |
| Alcohols having 11 to 16 carbons.....                           | 10         | 13.8       |
| Undecyl (branched).....   | 5.1        | 14.3       |
| Do.....   | 14.8       | 7.1        |
| Do.....   | 30         | -0.5       |
| Dodecyl (branched).....   | 7.6        | 8.6        |
| Tridecyl (branched).....  | 5          | 9.0        |
| Do.....   | 7.5        | 10.8       |
| Do.....   | 20         | 5.0        |
| Tetradecyl (branched).....                                      | 15         | 7.7        |
| Hexadecyl (branched).....                                       | 5          | 7.3        |
| Do.....   | 10         | 10.7       |
| Do.....   | 29.9       | 5.6        |
| Heptadecyl (branched).....                                      | 5          | 11.3       |
| Do.....   | 10.2       | 9.5        |
| Do.....   | 15         | 11.5       |
| Eicosyl (branched).....   | 7.4        | 2.9        |
| Do.....   | 15.1       | 4.8        |
| Do.....   | 39.2       | 3.0        |

Example 2: Determination of propionic acid content of rumen fluids was measured in vivo by withdrawing rumen samples from sheep fed a mixed corn and hay diet and analyzing the fluid. The propionic acid content when the feed contained two percent of tridecyl alcohol ethoxylated with 9.2 mols of ethylene oxide was 183% of that of a control experiment not containing the additive.

Example 3: Using the procedure of Example 2, the propionic acid content when the feed contained 1% of dodecyl alcohol ethoxylated with 3 mols of ethylene oxide was 161% of that of a control experiment not containing the additive. An in vitro experiment with the same additive showed a 7.2% increase in propionic acid content. The ethoxylated alcohols affect the rumen microflora and promote a more efficient use of the nutrients in conventional feeds. The ethoxylated alcohol is mixed with a ruminant feed by any conventional means. The ethoxylated alcohols are included in a ruminant feed as described earlier.

### Stimulating Growth of Desired Bacteria in Rumen

G.B. Garner (U.S. Patent 2,931,726; April 5, 1960; assigned to Norden Laboratories, Inc.) found that certain microorganisms are contaminants of the rumen in ruminant animals and Pseudomonas aeruginosa and Aerobacter aerogenes are present in rumen dysfunction



cases. Normally a regulating factor is present in rumen fluids which inhibits the growth of these undesirable organisms or contaminants and that this factor stimulates growth of desirable bacteria which produces the necessary enzymes for cellulose digestion. He discovered a factor or factors in fresh rumen fluid, its extracts, corn silage, sargo silage, feeds fermented by rumen microorganisms, material obtained from fermentation barrels inoculated with rumen microorganisms.

Example: One method of obtaining the growth regulator is to prepare it from rumen material taken from freshly slaughtered animals. This material is strained or pressed to remove the liquid. The liquid material is then centrifuged. The supernatant material in the centrifuging step is decanted and passed through an adsorbing column of an activated carbon at pH 3.5 to 4.0. The activated carbon may be of the type known under the trade name "Norit-A". The "Norit-A" adsorbs the active factor and permits the balance of the fluid to pass through. The adsorbing column is then eluted with 80% alcohol. The alcohol dissolves the factor from the adsorbing column. The alcoholic eluate may then be concentrated by removal of the alcohol by means of vacuum distillation to leave the product. The product thus obtained has the unique ability to suppress the growth of pathogenic or undesirable bacteria and at the same time, it stimulates the growth of desirable bacteria or those which produce enzymes for cellulose digestion. The extract is free of microorganisms and stable to autoclaving at a pH between 3.5 and 10.

The growth regulator may be used directly for treatment of various cases of rumen dysfunction where rumen contaminants are present. It has been demonstrated to be effective against Pseudomonas aeruginosa and Aerobacter aerogenes which have been isolated from rumen dysfunction cases. It may also be used as a premix concentrate additive to various prepared feeds for ruminants and other animals for improving feed utilization or efficiency.

### "Fermented Starter" Feed Supplement

C.O. Ensley (U.S. Patent 3, 150, 979; September 29, 1964) has developed a feed supplement for ruminants, 2 oz. of which is fed each day per head of cattle and 0.2 oz. per head of sheep. This does not provide basal food but appears to permit the enzymes present to step up the change of food into meat.

Example: The method of providing a feed supplement for ruminants includes:

(1) Forming a fermented starter which comprises (a) mixing 8 ounces of grass in the boot stage, 4 ounces of corn sugar, 16 ounces of starch, with sufficient water at 70° to 105°F. to bring about fermentation of the starter and (b) fermenting said starter in incubator 48 to 72 hours.

(2) Forming a fermented culture which comprises (a) mixing 9 ounces of fermented starter with 32 ounces of grass in the boot stage, 72 ounces of sugar, 128 ounces of starch, with sufficient water to bring about fermentation of the culture and (b) fermenting said culture 48 hours at 75° to 105°F.

(3) Dehydrating the culture may be accomplished in a dryer or by spreading a layer of the culture on screens placed one above the other and over and through which warm air is circulated. When dry, a culture is put through grinders to pulverize it. The means used should not heat the mixture.

(4) Mixing the finely ground fermented culture with a carrier which includes a plurality



of ingredients providing at least 18% of crude protein and 2% of crude fat, minerals and vitamins, to form a batch of the feed supplement to weigh out 2,000 pounds.

### Preservation of Bacterial Flora from Rumen Juice

In a process developed by L.G. Herman (U.S. Patent 2,938,794; May 31, 1960; assigned to Wilson and Company, Inc.) viable fungal spores and bacterial spores and/or cells are transferred from nonliquid, but moist, host media by means of a nontoxic crystalline carrying agent, preferably sucrose (sugar) crystals, to which the spores and cells adhere, and the freshly coated crystals are then quickly mixed, more or less immediately, with a dehydrating or desiccating agent in particulate form so that the spore and/or cell-coated crystals retain their single crystalline characteristics.

The method for the harvesting, drying and preservation of the bacterial flora from rumen juice finds special usefulness for the preparation of a medicinal product for calves and for convalescent cattle and other species of the ruminant groups to restore lost rumen flora. The bacterial flora to be preserved is obtained as follows. The paunch contents of healthy steers, heifers, sheep or other ruminant, preferably as it is taken from the animal, is passed through a screen, suitably of a 15 to 40, preferably 20, mesh size screen, to remove from the rumen juice the coarser material such as corn, hay, grass and other undigested food and the larger particles of inert solid material. The material held back, on the screen is pressed and squeezed on the screen, as by hand, to remove additional amounts of rumen juice, particularly that which adhered to the surfaces of the screened material. The screened material is discarded. The rumen juice with its content of bacterial flora and unclassified solids material which passed through the screen is centrifuged, suitably in a Sharples clarifying bowl type centrifuge or other type of centrifuge, running at about 30,000 to 60,000 rpm preferably 50,000 rpm.

The resulting sediment, rumen concentrate, containing the protozoa and bacteria thrown out of the juice during centrifugation is removed from the machine. It is in a semimoist more or less plastic state. At normal room temperatures of 70° to 80°F. it has the consistency of butter. The rumen concentrate is now mechanically mixed with a water-soluble crystalline carrier, preferably sucrose crystals, the resulting bacteria-coated crystals are dehydrated by being mixed with desiccant granules, and then the desiccant granules are removed as by sieving. The mixing of the rumen concentrate is effectively accomplished in a conventional Buffalo mixer running at full speed for about 6 to 12 minutes. The desired product of this mixing operation is a nonsyrupy homogeneous brown mass, much like commercial brown sugar. Varying amounts of rumen concentrate with respect to the sucrose crystals may be used to obtain this homogeneous brown mass, say 15 to 25 parts by weight of sucrose crystals to 1 part by weight of rumen concentrate. The preferred ratio is 20 parts by weight of the concentrate to 1 part by weight of the sugar.

When the mixing of the rumen concentrate and of the sugar is completed there is then added thereto in the mixer a quantity of desiccant granules sufficient to dehydrate the mass. In the case of a mixture of 1 part by weight of rumen concentrate and 20 parts by weight of sugar, 40 parts by weight of silica gel crystals are adequate to effect the desired dehydration. Mixing of the desiccant granules into the mass in the mixer is continued until dry dusty material begins to separate from the mass, at which point mixing is stopped. At this stage the



product is apparently dry and free flowing. The substantially dry mixture is removed from the mixing apparatus and the desiccant granules are removed as by sieving, all as described above.

The resulting dehydrated product is free flowing and easily handled or measured for addition to feeds. It consists of sugar crystals coated with the bacterial flora and other material in the rumen concentrate. The rumen concentrate including the bacterial flora is imprisoned on the surfaces of the sugar crystals by a thin film of sugar in a glassy state. This thin binding film is obtained by the wetting of the sugar crystal surfaces on contact with the moist rumen concentrate and the subsequent dehydration thereof by the desiccant granules.

The bacteria-coated sugar crystals may be fed in the dry, powder state to calves and convalescent cattle, but preferably the powder is preliminarily compacted into pellet form. If desired, this dry product, in powder or pellet form, may be mixed with skim milk, other conventional form of calf and cattle feeds, vitamins, etc. and used as a food supplement. When the product is fed to calves or cattle, solution of the product in the paunch juice is almost instantaneous and both the sugar and the organisms are rapidly distributed through the rumen contents. In the processing of the rumen contents to obtain the rumen concentrate and in the subsequent treatment thereof with sugar and desiccant to obtain the dry, free flowing product of the present process, it is desirable to maintain the temperature of the material undergoing treatment above about 70° to 75°F., so that the workability of the material is not impaired, and below about 105°F., so that the material is not deleteriously affected.

When the desiccant granules are mixed into the mixture of the bacterial flora and sugar in the mixer there is a tendency of the mixture to rise to about 85° to 95°F., occasionally up to about 100°F. It is preferred that the temperature of the mixture in the mixing machine be controlled, as by cooling coils, so that the temperature thereof remains below about 105°F. It is imperative that the temperature of the materials at all stages of processing be maintained below incipient pasteurizing temperatures of 127° to 130°F.

### Rumen Microorganisms Plus Lactic Ferment

J.P. Mecho and E.G. Sicilia (U.S. Patent 3,243,299; March 29, 1966; assigned to Pronit Internacional SA, Spain) provide a preparation of the microflora of the rumen and the reticulum of ruminant animals which is capable of being fed to monogastric animals and which will not be adversely affected by the stomach acidity of monogastric animals, and enable them to digest cellulosic feeds. It has been found that the microflora of the rumen and reticulum of ruminants can be made to continue to live and to multiply in an environment with a pH between 4.5 and 5.0 by isolating said microflora and incorporating with the living microflora a quantity of a lactic ferment sufficient to protect the microflora against the acidity of the monogastric stomach, namely a pH between about 4.5 and 5.0. By lactic ferment is meant a preparation, preferably in dry powder form, of acidophile bacilli of a type which are resistant to and capable of growth in an environment having a pH between about 4.5 and 5.0, i.e. which are capable of growth in monogastrics. These organisms include for example, lactic acid bacteria of the Lactobacillus bulgaricus and Lactobacillus acidophilus types.

The stomach contents, containing the microflora, are withdrawn using conventional techniques and avoidance of contamination, and are transferred to a mixing tank, and sufficient bran is



added to permit about 60% of moisture to remain in the contents. Toluene may be added as a preservative. The mixture is cultured for about 24 hours at room temperature. A small proportion of the fermented mix is then employed to inoculate a cultural broth, and the remainder of the mix is dried as described below.

The culture portion of the mix is cultured under anaerobic conditions using any suitable broth customarily employed for growing microorganisms or yeasts, and which contains nitrogen, carbohydrates and minerals. Thus, there may be used a broth made from 500 grams of lean minced beef soaked in water, to which is added 1% of Witte's peptone, and 0.5% sodium chloride; glucose, tryptone, and potassium acid phosphate may also be added. The broth may also include molasses, urea or ammonium salts, and a substrate such as sawdust. The pH is maintained between about 6.8 and 7.2 to simulate conditions in the rumen. Culture takes place at a temperature between about 35° and 40°C., preferably at 35°C. for 24 to 48 hours, whereupon the broth is centrifuged to lower its moisture content to about 30 to 40% by weight, and to provide a product comprising live microflora and sawdust residue.

The remainder of the rumen contents-bran mixture previously described is divided into three parts which, for convenience, may be three equal parts. The first portion is dried under vacuum at a temperature between about 35° and 40°C. to obtain a product comprising live microflora and having a moisture content of about 10% to 12%. The drying should take the shortest possible time, 3 to 6 hours, and the degree of vacuum need be only slight, for example, about 50 mm. of vacuum. The second portion is dried at a temperature between about 50° and about 57°C., preferably at 55°C. at atmospheric pressure, for a period of approximately 8 hours. Under these conditions, bacteria are destroyed and autolysis takes place, yielding a meal containing about 6% moisture which is rich in enzymes of the rumen microflora, such as, for example, aminases, proteases, cellulases, and hemicellulases.

The third portion is dried at about 70°C. for about 1 hour, the object being to obtain spores of the rumen bacteria. These, by a process of natural selection, will be the stronger spores, which are capable of reproduction and retain more activity. The portion is dried to about 5% moisture content. It will be understood that the relative amounts of the aforesaid three portions may be varied as desired, and that the conditions set forth may also be subject to considerable variation, depending upon the type of organisms and other process conditions. The rumen contents and the reticulum contents may be treated separately as desired. The foregoing materials are admixed to provide a preparation containing live rumen and/or reticulum organisms as the active ingredient thereof, and containing about 10% moisture.

The foregoing materials are admixed to provide a preparation containing live rumen and/or reticulum organisms as the active ingredient thereof, and containing about 10% moisture. To the mixture of microflora and dried materials there is added a suitable amount of lactic ferment. There is also added yeast, preferably brewers yeast (*Saccharomyces*) to aid in the growth of the microflora and to give volume to the preparation, and to aid in the synthesis of proteins and vitamins in the monogastric animal. There also are added small amounts of mineral nutrients, including potassium iodide, sulfur (in the form of flowers of sulfur) and cobalt sulfate.

Example: 500 kg. of the contents of the rumen and the reticulum of a freshly slain cow were extracted, and treated as described previously, by culturing with bran for 24 hours, removing



## Ruminant Feeds

a small portion for further broth culture, dividing the remainder of the bran mixture into three equal portions. The broth was cultured for 36 hours at pH 7 and at 35°C., and the broth was centrifuged and the product set aside. The first portion of bran mixture was dried at 50 mm. vacuum at 37°C. for 3 hours. The second portion was dried at 55°C. for 8 hours at atmospheric pressure. The third portion was dried at 70°C. for 1 hour. The dried portions and the culture were admixed with lactic ferment, and yeast, and mineral materials, to yield a product having the following formula:

|  |              |     |
|--|--------------|-----|
| Rumen and reticulum microorganisms     | -----kg--    | 50  |
| Top yeast containing max. 12% moisture | -----kg--    | 350 |
| Dried yeast                            | -----kg--    | 100 |
| Lactic ferments in powder form         | -----kg--    | 1   |
| Potassium iodide, 50% solution         | -----grams-- | 20  |
| Sulfur                                 | -----do--    | 10  |
| Cobalt sulfate                         | -----do--    | 2.5 |

The foregoing microflora preparations are suitable as additives for admixture with vegetable protein materials, other nitrogenous materials and mineral ingredients, to provide feed concentrates. The feed concentrates may, in turn, be further admixed with corn, oats, and cellulosic feed ingredients to provide a feed composition suitable for feeding monogastrics. In the concentrates, the additive described above will be designated for convenience by its trademark "Pronit". It will be seen from the formula that animal proteins, such as fish meal, have been completely replaced by vegetable proteins. The Pronit increases the utilization of the vegetable proteins and makes possible to a limited extent, the assimilation of the urea, converting nonprotein nitrogen into assimilable protein within the stomach of the monogastric.

|   | Percent      |
|---|--------------|
| Soya or other plants with vegetable protein | 66.25        |
| Urea  | 12.50        |
| Pure Pronit                                 | 2.50         |
| Dicalcium phosphate                         | 11.25        |
| Calcium carbonate                           | 5.00         |
| Fats  | 2.50         |
|   | <hr/> 100.00 |

### Urea-Phosphoric Acid-Glycerine Additive

W. J. Motzel (U.S. Patent 3,185,572; May 25, 1965) has prepared compositions useful as additives to drinking water and feed for herbivorous animals, especially ruminants which stimulate and intensify bacteria present in the rumen.

The following composition has been found to be especially suited and advantageous: 50% of urea, 20% of phosphoric acid of 75% strength, and 30% of glycerine. The compositions are added to the drinking water of the animals in amounts equivalent to 25 grams of the composition for each drink per animal, or it may also be added to animal feeds, such as bran, chaff and turnips. Animals fed this additive have a fine smooth skin and a higher protein fat ratio.

### 8-Hydroxyquinoline Derivatives

Farm animals and particularly cattle and cows, which feed extensively on grass and hay, are

subject to certain digestive upsets and disorders due to bacterial overgrowth, especially of the paratyphoid, coli, salmonella and aerobacter groups of intestinal bacteria, diarrhea and excessive gas formation. Bacteria overgrowth is equivalent to a low degree intestinal infection which is capable of destroying valuable nutrients and impeding proper digestion and assimilation of food and consequently of interfering with absorption through the intestinal wall with resulting waste of nutrients.

W. Rosenthal (U.S. Patent 3,021,216; February 13, 1962) found that by combining with feed or mash an 8-hydroxyquinoline derivatives, the animals are rendered free or substantially free from the disadvantages set forth above, thus promoting their health and growth as well as weight increase and milk production and better feed efficiency. The 8-hydroxyquinoline derivative is added to and thoroughly mixed with the mash or other conventional nutrient feed in such proportions that when the animals ingest their normal daily ration they at the same time ingest from about 10 mg. to 10 grams per day per 1,000 pounds of body weight of the 8-hydroxyquinoline derivative. Typical and illustrative derivatives of 8-hydroxyquinoline are:

- 5-chloro-7-iodo-8-hydroxyquinoline
- 5-chloro-7-bromo-8-hydroxyquinoline
- 5,7-dichloro-8-hydroxyquinoline
- 5,7-dibromo-8-hydroxyquinoline
- 7-iodo-8-hydroxyquinoline
- 7-iodo-8-hydroxyquinoline-5-sulfonic acid
- 7-iodo-8-hydroxyquinoline-5-sulfonic acid sodium salt
- 7-iodo-8-hydroxyquinoline-4-sulfonic acid calcium salt
- 7-iodo-8-hydroxyquinoline-5-sulfonic acid copper salt

The salts may be either water-soluble salts or water-insoluble salts, the latter being preferred, however, since their property of water-insolubility is an advantage in that the additive does not leach out of the feed. The water-soluble salts are advantageous where it is desired, for example, to add the same to the drinking water of the animal.

## OTHER GROWTH PROMOTERS

### Mono-C-Substituted Triazoles

The process of R.G. Eggert and W.T. Akers, (U.S. Patent 3,148,068; September 8, 1964; assigned to American Cyanamid Company) depends on the discovery that a class of compounds, toxic to weeds, namely, certain C-monosubstituted 1,2,4-triazoles, are not only nontoxic to ruminants in small amounts but produce a marked increase in growth in these animals. The triazoles may be used as the only additive or they may be combined with a female sex hormone-like compound, such as diethylstilbestrol, which further enhances the growth of fattening cattle. Among the mono-C-substituted triazoles which are most effective is ordinary 3-amino-1,2,4-triazole, which is one of the best; 3-alkanoylamino-1,2,4-triazoles, such as 3-acetylamino, 3-propionylamino, 3-butyrylamino, etc.; and 3-furfurylideneamino-1,2,4-triazole. It has been found that these beneficial growth promoting effects can be accomplished in fattening cattle when as little as 5 mg. per pound of total feed, and as much as 120 mg.



## Ruminant Feeds

per pound of feed are used (preferred range 10 to about 40 mg. ).

Example: Feed was prepared as follows:

|  | Percentage   |
|--|--------------|
| Coarse cracked corn .....              | 66.8         |
| Crimped oats .....                     | 15.0         |
| Soybean oil meal .....                 | 6.0          |
| Blackstrap molasses .....              | 4.0          |
| Dehydrated alfalfa meal .....          | 5.0          |
| Ground limestone .....                 | 1.0          |
| Steamed bonemeal .....                 | 1.0          |
| Iodized salt .....                     | 1.0          |
| Trace mineral mix <sup>1</sup> .....   | 0.1          |
| Vitamin A and D mix <sup>2</sup> ..... | 0.1          |
| <b>Total</b> .....                     | <b>100.0</b> |

<sup>1</sup> Furnished the following amounts of trace minerals in the ration: manganese, 60 p.p.m.; iron, 20 p.p.m.; iodine, 1.2 p.p.m.; copper 2 p.p.m.; zinc, .06 p.p.m.; and cobalt, .2 p.p.m.  
<sup>2</sup> Furnished 4,500 I.U. of vitamin A and 900 I.U. of vitamin D per pound of ration.

The food was divided into three lots, to one of which nothing is added, to one 20 mg. of 3-amino-1,2,4-triazole compound and to a third 40 mg. of 3-amino-1,2,4-triazole per pound of feed. Steers were fed for 56 days on the above feeds, the average ingestion being 15 pounds per day. This was supplemented with timothy-alfalfa hay which was given ad libitum.

The effects of the feedings are shown below,

|                         | Milligrams of 3-Amino-triazole per pound of feed |        |        |
|-------------------------|--|--------|--------|
|                         | 0  | 20 mg. | 40 mg. |
| Av. Initial Weight..... | 762  | 750    | 793    |
| Av. Final Weight.....   | 877  | 907    | 960    |
| Av. Daily Gain.....     | 2.07   | 2.80   | 2.98   |

It will be apparent that at the 20 mg. per pound of feed, the gain was 35% and at 40 mg. per pound of feed was about 44% over that obtained with the basal ration.

### 3,3-Bis (4-Hydroxy-Phenyl) Pentane

The administration of 3,3-bis (4-hydroxyphenyl) pentane to animals in physiologically adequate amounts exerts a growth-promoting action according to G.B. Kline and R.Q. Thompson, (U.S. Patent 3,098,745; July 23, 1963; assigned to Eli Lilly & Company). The 3,3-bis (4-hydroxyphenyl) pentane can be administered or combined with any of the usual animal feeds or feed supplements in such amount that it is present in the feed or supplement in an amount of about 1 to 20 grams per ton, preferably about 2 to 10 grams per ton. 3,3-bis (4-hydroxyphenyl)-pentane can conveniently be administered to the growing animal in dosages based on body weight. It is preferred to employ a dosage ranging from about 0.1 to about 5 mg. of 3,3-bis(4-hydroxyphenyl) pentane per 100 pounds of body weight over each 24 hour period.

TABLE 1: LAMB BASAL RATION A

| <u>Ingredient</u>                                | <u>Percent of<br/>Ration</u> | <u>Pounds Per<br/>Ton</u> |
|--|------------------------------|---------------------------|
| Corn, yellow                                     | 31.00                        | 620                       |
| Cobs, corn                                       | 37.50                        | 750                       |
| Alfalfa meal, dehydrated 17%                     | 11.00                        | 220                       |
| Soybean oil meal, solvent extracted dehulled 50% | 7.75                         | 155                       |
| Cottonseed meal, solvent extracted 41%           | 1.75                         | 35                        |
| Urea, feeding grade                              | 0.52                         | 10.50                     |
| Distillers' dried grains with solubles (corn)    | 1.75                         | 35                        |
| Molasses, cane                                   | 7.50                         | 150                       |
| Dicalcium phosphate, feed grade                  | 0.53                         | 10.50                     |
| Calcium carbonate                                | 0.35                         | 7                         |
| Salt (NaCl)                                      | 0.35                         | 7                         |
| Minerals (trace) <sup>1</sup>                    | 0.06                         | 1.125                     |
| Vitamin D <sub>2</sub> premix <sup>2</sup>       | 0.05                         | 1                         |
| Total  | 100.11                       | 2,002.125                 |

<sup>1</sup> See footnote <sup>1</sup> under Table 2.<sup>2</sup> See footnote <sup>2</sup> under Table 2.

TABLE 2: LAMB BASAL RATION B

| <u>Ingredient</u>                                | <u>Percent of<br/>Ration</u> | <u>Pounds Per<br/>Ton</u> |
|--|------------------------------|---------------------------|
| Corn, yellow                                     | 56.00                        | 1,120                     |
| Cobs, corn                                       | 20.00                        | 400                       |
| Alfalfa meal, dehydrated 17%                     | 3.00                         | 60                        |
| Soybean oil meal, solvent extracted dehulled 50% | 6.50                         | 130                       |
| Cottonseed meal, solvent extracted 41%           | 1.50                         | 30                        |
| Urea, feeding grade                              | 0.45                         | 9                         |
| Distillers' dried grains with solubles (corn)    | 1.50                         | 30                        |
| Molasses, cane                                   | 10.00                        | 200                       |
| Dicalcium phosphate, feed grade                  | 0.45                         | 9                         |
| Calcium carbonate                                | 0.30                         | 6                         |
| Salt (NaCl)                                      | 0.30                         | 6                         |
| Minerals (trace) <sup>1</sup>                    | 0.06                         | 1.125                     |
| Vitamin D <sub>2</sub> premix <sup>2</sup>       | 0.05                         | 1                         |
| Total  | 100.11                       | 2,002.125                 |

<sup>1</sup> CCC Trace Mineral Premix contains: 12.20 percent manganese as manganese sulfate, 0.38 percent iodine as potassium iodide, 0.26 percent cobalt as cobalt sulfate, 9.60 percent iron as ferrous sulfate and ferrous carbonate and red iron oxide (for color), 0.73 percent copper as copper carbonate, 5.00 percent zinc as zinc sulfate and zinc oxide, and 6.35 percent calcium as calcium carbonate.

<sup>2</sup> Each pound contains 500,000 USP units vitamin D<sub>2</sub>. Premix made by adding one pound of 64,000,000 USP units per pound irradiated yeast to 125 pounds of soybean feed.



## Ruminant Feeds

Example: This example compares the effects on growth rate and feed efficiency of lambs receiving a nutritionally adequate per se basal ration (negative control), and lambs receiving said ration containing diethylstilbestrol (positive control), with lambs receiving a ration containing the growth-promoting ingredient of this process, 3,3-bis(4-hydroxyphenyl) pentane.

The feeding procedure employed an experimental design of the block design type, utilizing a randomization method of allotment. The lambs employed were eight month, Texas white face wethers. (Each lamb was fed for a two week conditioning period and then weighed, identified, and drenched with two ounces of phenoarsenate.) These lambs were divided into five lots of ten lambs each, and one lot containing nine lambs. In the negative control, two lots of ten lambs each were fed basal ration B for six days and basal ration A for the duration (see Tables 1 and 2 for the composition of basal rations A and B). The positive control comprised two lots of nine and ten lambs each, which were fed basal ration B plus 2 mg. per head per day of diethylstilbestrol for six days, and basal ration A plus 2 mg. per head per day for the duration of the test period. Additionally, two lots of ten lambs each were fed basal ration B plus 2 mg. per head per day of 3,3-bis (4-hydroxyphenyl) pentane for six days, and basal ration A plus 2 mg. per head per day of 3,3-bis (4-hydroxyphenyl) pentane for the duration. The total test period was 42 days. The results of the experiment are set forth in Table 3 below.

**Daily Gain and Feed Efficiency Data**  
**3,3-BIS(4-HYDROXYPHENYL)PENTANE**

|                                     | Number<br>of<br>Animals | Avg.<br>Daily<br>Gain,<br>Lbs. | Feed<br>Effi-<br>ciency <sup>4</sup> |
|-------------------------------------|-------------------------|--------------------------------|--------------------------------------|
| Negative Control <sup>1</sup> ..... | 20                      | 0.51                           | 13.1                                 |
| Positive Control <sup>2</sup> ..... | 19                      | 0.54                           | 14.1                                 |
| Test Material <sup>3</sup> .....    | 20                      | 0.57                           | 14.2                                 |

<sup>1</sup> Basal ration.

<sup>2</sup> Basal ration plus 2 mg. diethylstilbestrol.

<sup>3</sup> Basal ration plus 2 mg. 3,3-bis(4-hydroxyphenyl)pentane.

<sup>4</sup> Weight gained per unit of feed consumed, expressed as percentage.

Thus, in the example, 3,3-bis (4-hydroxyphenyl) pentane in feed at levels of 2 mg. per head per day improved rate of gain 11.8%, when compared to the negative control basal ration (0.57 vs. 0.51 pound), and was 5.6% better than diethylstilbestrol (0.57 vs. 0.54 pound). Feed efficiency was improved 8.4%, when compared to the negative control (14.2 vs. 13.1), and was better than diethylstilbestrol (14.2 vs. 14.1).

### 2-Mercaptoimidazole

The following two processes are concerned with the administration of 2-mercaptoimidazole to meat producing ruminants for promoting growth and feed utilization. One limiting factor which has been encountered in the use of 2-mercaptoimidazole compounds with ruminants is that the growth promotant response can be obtained only for relatively limited periods of time. More specifically, the maximal response has usually been obtained in the first thirty days, while the response diminishes during the next thirty days. Since beef cattle are commonly fed for periods of 100 to 150 days, it would be desirable to provide means for extending the length of time during which an appreciable growth promotant effect can be obtained with a 2-mercaptoimidazole compound.



W. Burroughs discovered (U.S. Patent 3,210,194; October 5, 1965; assigned to Iowa State University Research Foundation, Inc.), that the quantity of vitamin A received by the ruminants can markedly affect the growth promotant response from a 2-mercaptoimidazole compound. If the quantity of vitamin A in the diet is excessive the response obtained with the 2-mercaptoimidazole compound will be reduced. It has been found desirable to employ at least two different feeding regimens in successive time periods. More specifically, during the first phase of the 2-mercaptoimidazole administration the diet for the ruminants should contain an average of less than 1,500 International Units (I.U.) of vitamin A activity per pound of ration. It will be understood that this is on a total ration basis.

The results are still further improved when the vitamin A level in the first phase is limited to less than 800 I.U. per pound of ration. Since the natural feed materials included in the diet of the animals will normally supply a considerable amount of vitamin A activity as carotene (e.g., B-carotene) the natural vitamin A precursor (sometimes referred to as pro-vitamin A), it will normally not be either necessary or desirable to add vitamin A to the feed materials being administered to the ruminants during the initial feeding period. In fact, it will usually be necessary to limit the intake of high carotene ration ingredients, such as natural hays and fodders, to assure that the total vitamin A activity in the complete ration is below the limit specified.

Since immature, growing beef cattle and sheep eat an amount of feed in 24 hours corresponding to approximately 3% of their body weight, the foregoing vitamin A levels can also be expressed in terms of animal body weight. For example, the vitamin A level for beef cattle and sheep during the first phase of the 2-mercaptoimidazole administration is preferably limited to less than 2,400 I.U. of vitamin A per 100 pounds of body weight. In all cases, the diet for the animals during the first phase should contain less than 4,500 I.U. of vitamin A per 100 pounds of body weight per 24 hours.

The first phase of the 2-mercaptoimidazole administration, as described above, should preferably extend for at least 30 days. By way of specific illustration, the first phase could extend for a period of from 45 to 60 days, while the second phase lasted for a period of from 30 to 90 days. During the second phase of the 2-mercaptoimidazole administration, the carotene level in the feed appears to be of lesser importance than during the first phase. After the 2-mercaptoimidazole compound has been administered for from 30 to 60 days, the conversion of carotene to vitamin A by the animal seems to be largely blocked. This makes it easier to control the amount of vitamin A below that which would interfere with the 2-mercaptoimidazole response, but at the same time it may tend to result in a condition of vitamin A deficiency where the animal is largely dependent on carotene as the vitamin A source.

Consequently, in the second phase of the 2-mercaptoimidazole administration, the ruminants are fed a diet containing supplementary vitamin A in the form of true vitamin A in an amount ranging from 100 to 1,500 I.U. per pound of ration on a total ration basis, (preferred 200 to 1,000 I.U.). Expressed differently, the quantities of vitamin A received by the animals during the second phase of the 2-mercaptoimidazole administration should be kept within the range from 300 to 4,500 I.U., and preferably from 600 to 3,000 I.U. of vitamin A per 100 pounds of body weight per 24 hours. The 2-mercaptoimidazole compound can be selected from the group consisting of 2-mercaptoimidazole and 1-alkyl 2-mercaptoimidazoles wherein the alkyl group contains from one to five carbon atoms. One preferred compound is



1-methyl 2-mercaptoimidazole. The quantity of the 2-mercaptoimidazole compound to be employed will be substantially in accordance with prior practice. Speaking generally, at least 10 mg. but not over 150 mg. of the 2-mercaptoimidazole compound should be employed per 100 pounds of body weight per 24 hours, (optimum amounts 25 to 100 mg.). For beef cattle of the size which are normally being fattened for market, a dose of from 200 to 1,000 mg. of the 2-mercaptoimidazole compound per animal per 24 hours can be used, while for sheep the dose will be correspondingly smaller, for example, from 20 to 100 mg. of the 2-mercaptoimidazole compound per animal per 24 hours. As a matter of convenience, the quantity of the 2-mercaptoimidazole compound administered can be the same during the first and second feeding period, although ideally the quantity should be increased in proportion to the increase in body weight of the animals. It will be convenient to administer the 2-mercaptoimidazole compound as an ingredient in a beef cattle or sheep protein feed supplement.

Such supplements are commonly fed to beef cattle in amounts of 1 to 2 pounds per day while sheep may receive from 0.1 to 0.2 pound per day. Depending somewhat on the amount fed, said protein feed supplements for use in accordance with this process can contain from 150 to 1,000 mg. of 2-mercaptoimidazole compound per pound. If desired the 2-mercaptoimidazole compound can be included in a complete ration for the beef cattle or sheep. Usually the dosages can be computed with sufficient accuracy on the assumption that the animals will eat about 3% of their weight per day.

For use in practicing the second phase of the method of this process, the protein feed supplement or complete ration should contain supplemental vitamin A as true vitamin A. Preferably, from 6 to 120 I.U. of vitamin A is present per milligram of the 2-mercaptoimidazole compound. In addition to the 2-mercaptoimidazole compound and the vitamin A, the protein feed supplement will contain feed ingredients having a relatively high protein content, such as soybean meal, cottonseed meal, etc. It will be understood, however, that the 2-mercaptoimidazole compound and the vitamin A may be combined with other feed ingredients, either as a premix, a feed supplement, or a complete feed ration.

In this process, W. Burroughs (U.S. Patent 3,248,222; April 26, 1966; assigned to Iowa State University Research Foundation, Inc.) found that thyroxine-active substances when administered in controlled amounts are capable of enhancing the effectiveness of 2-mercaptoimidazole compounds. This is believed to be a synergistic effect since the increase in rate of weight gain and in feed efficiency cannot be accounted for by the individual effects of the 2-mercaptoimidazole compound and thyroxine-active substance. In order to achieve the desired results, however, it is necessary to limit the amount of thyroxine-active material to from 0.0002 to 0.0075 mg. of thyroxine (or a quantity providing this amount of thyroxine activity) per mg. of the 2-mercaptoimidazole compound. Preferably, the quantity of thyroxine-active substance should provide from 0.0005 to 0.0025 mg. of thyroxine activity per mg. of 2-mercaptoimidazole compound. The amount of 2-mercaptoimidazole employed is the same as that described above.

It will usually be desirable to administer the combination of the thyroxine-active material and the 2-mercaptoimidazole compound for at least 30 days, and preferably for a period of at least 60 days. Where it is desired to administer to cattle the combination of the 2-mercaptoimidazole compound and the thyroxine-active material, either with or without an estrogenic substance, for



periods of longer than 60 days, it is preferable to limit the intake of vitamin A activity during the first 60 days, as described in the previous page, and then after the first 60 day period to incorporate a controlled amount of true vitamin A in the ration. At a later stage in the feeding of beef cattle and sheep it will probably be desirable to employ a feed material continuing from 0.0002 to 0.0075 mg. of thyroxine activity together with 2 to 450 I.U. of true vitamin A per mg. of the 2-mercaptoimidazole compound. A preferred formulation will contain from 6 to 120 I.U. of the vitamin A and from 0.0005 to 0.0025 mg. of thyroxine activity per mg. of the 2-mercaptoimidazole compound.

Example: The powder form of 700 g. of 1-methyl 2-mercaptoimidazole and 100 g. of a thyroprotein containing 1% thyroxine activity is thoroughly blended in approximately 8 pounds of wheat middlings to make 10 pounds of a first premix. The premix thus compounded will contain 70 g. of the 1-methyl 2-mercaptoimidazole compound and 10 g. of thyroprotein having 0.1 g. of thyroxine activity per pound of the product. This premix product prepared as described above can then be used to prepare a first feed ration or protein supplemental feed for administration to beef cattle or sheep during the first phase of a feeding regimen comprising a feeding period of 30 to 60 days. For example, 10 pounds of the premix can be mixed with 1,990 pounds of protein supplemental ingredients which are essentially devoid of carotene and vitamin A. Such ingredients are principally composed of vegetable protein sources such as soybean meal, cottonseed meal, linseed meal, wheat middlings, etc.

After the premix has been thoroughly distributed throughout the ingredients, a ton of protein supplemental feed utilizable in the first feeding period of this process will be obtained. This material will contain 350 mg. of 1-methyl 2-mercaptoimidazole and 0.5 mg. of thyroxine activity per pound and the material will be essentially devoid of vitamin A or vitamin A activity in the form of carotene.

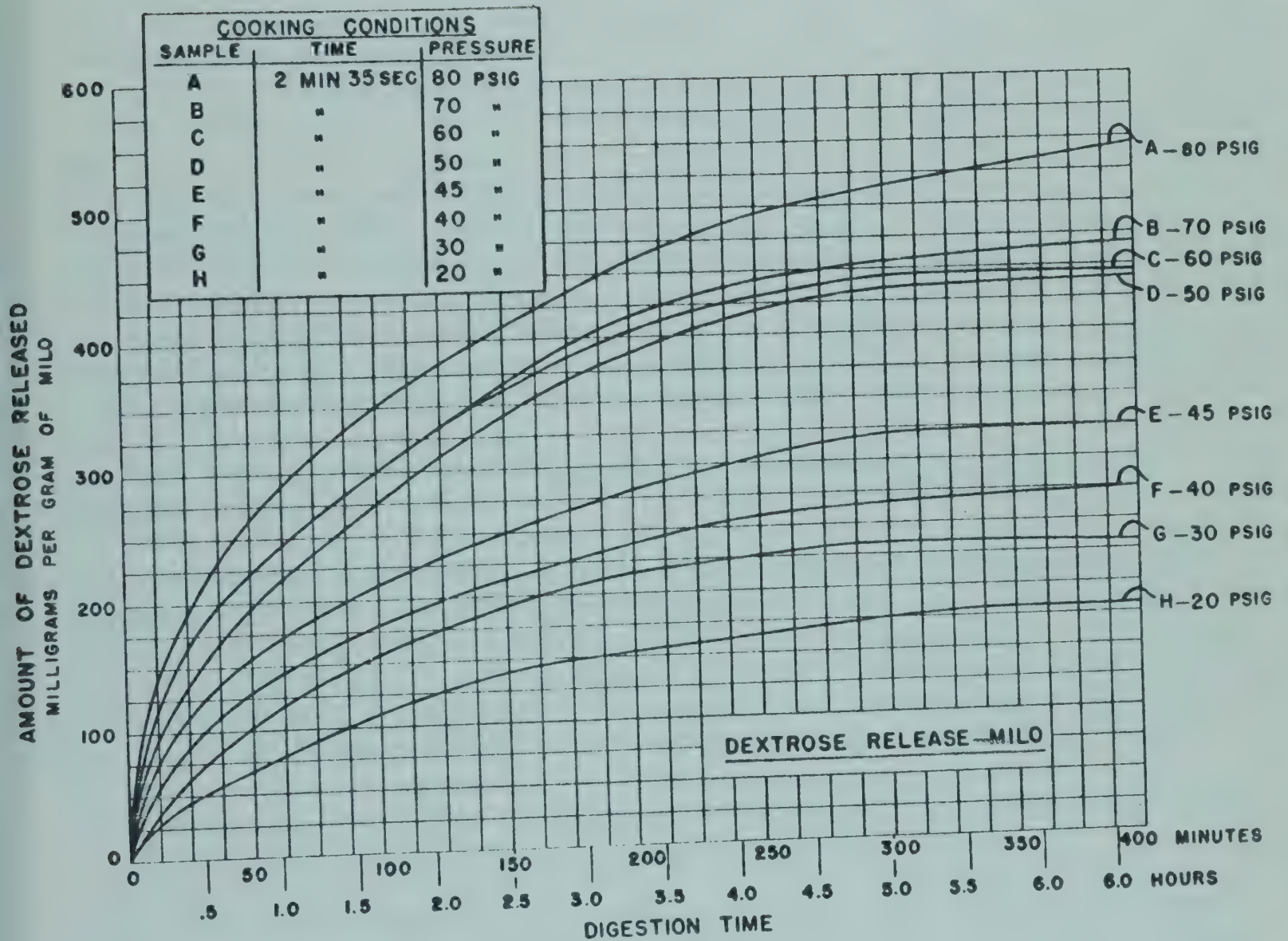
During the second feeding period extending over an additional period of 30 to 60 days, the feed ration or protein supplement fed to beef cattle or sheep is prepared similar to that used in the first feeding period except that a controlled amount of true vitamin A is also incorporated. For example, a second protein supplemental feed is fed during this second feeding period by first preparing a second premix. 700 g. of 1-methyl 2-mercaptoimidazole, 100 g. of a thyroprotein containing 1% thyroxine activity and 8 million I.U. of vitamin A are thoroughly dispersed on about 8 pounds of soybean meal to make 10 pounds of a second premix. This 10 pounds of second premix is thoroughly mixed with 1,990 pounds of other protein supplemental ingredients as described above to make one ton of protein supplemental feed suitable for feeding during this second feeding period.

## INCREASING DIGESTIBILITY OF STARCHES

In many uses of cereal grains, the carbohydrate material in the grain is acted on by enzymes to convert the carbohydrates to dextrose. In the feeding of livestock, for example, the enzymes secreted by the organisms in the rumen and intestine of the animal act on the carbohydrates in cereal grains to convert them to a readily metabolizable form. The ability of the enzymes to convert carbohydrate to the desired dextrose can be increased by treating the cereal before it is fed to the animal. These processes deal with treatment of grain for



FIGURE 11.1: PROCESSING CEREAL GRAINS FOR BETTER UTILIZATION



Source: F.D. Hickey; U.S. Patent 3,336,137; August 15, 1967

better digestibility and thereby more efficient utilization by animals.

### Processing Cereal Grains

F.D. Hickey (U.S. Patent 3,336,137; August 15, 1967; assigned to FMC Corporation), provides a process of treating cereal grains to condition them so that the carbohydrates therein can be more efficiently converted to dextrose when acted on by enzymes secreted by organisms in the rumen and intestines of animals.

Example: In accordance with this process, cereal, such as milo and corn having normal moisture content, is cooked in a saturated steam atmosphere at a pressure such that the large starch molecules are broken down into small molecules which are relatively easily digested. For certain cereals the cooking can be done at a pressure in the range of 40 to 100 psig for from 15 seconds to 5 minutes. The term "normal moisture content" will be used hereinafter to mean the amount of moisture in the cereal when it is exposed to the usual ambient weather conditions. For corn and milo, such normal moisture content may be in the range from 9 to 13%, although it may be less in dry climates. After cooking, the cereal is rolled and flaked to subject it to shear stresses and disorganize the tissue structure while it is relatively hot. When this cooked and rolled cereal is cooled, it is in a condition such that enzyme-catalyzed hydrolysis can convert considerably more carbohydrates to dextrose, in any given period of time, than they could convert if the cereal had merely been steamed or had been cooked at a pressure that did not cause the breakdown of the large starch molecules.

The exceptional carbohydrate conversion rate obtained when cereals are processed under predetermined conditions has been demonstrated in tests where small samples of cereal were cooked at different pressures and then rolled to put the cooked cereal into flake form. When these flakes are ground and subjected to the action of enzymes, the amount of dextrose that is formed during each time interval is much greater with flakes formed from cereals that have been cooked at pressures in the range of from 40 to 100 psig than the amount formed when the cereal is merely steamed or is cooked at pressures below 40 psig. Thus, for the cereals used in these tests, the breakdown of the starch molecules appears to start to take place at an exceptional rate at approximately 40 psig. This is illustrated graphically in Figure 11.1.

### Pretreatment of Starch Component of Mixed Feedstuff

P.A. Singer (U.S. Patent 3,438,780; April 15, 1969; assigned to Allied Mills, Inc.) has developed a process in which the starch components of a feedstuff are preconditioned and tempered for improved rupturing of starch cells, so that the starch bearing material is better utilized during animal consumption.

Example: This example illustrates the preparation of a cattle or dairy feed. The ingredients for the cattle or dairy feed are as follows:

|             | Pounds |
|-------------|--------|
| Ground corn | 47.00  |
| Ground milo | 10.00  |
| Hominy feed | 10.00  |

(continued)



## Ruminant Feeds

(continued)

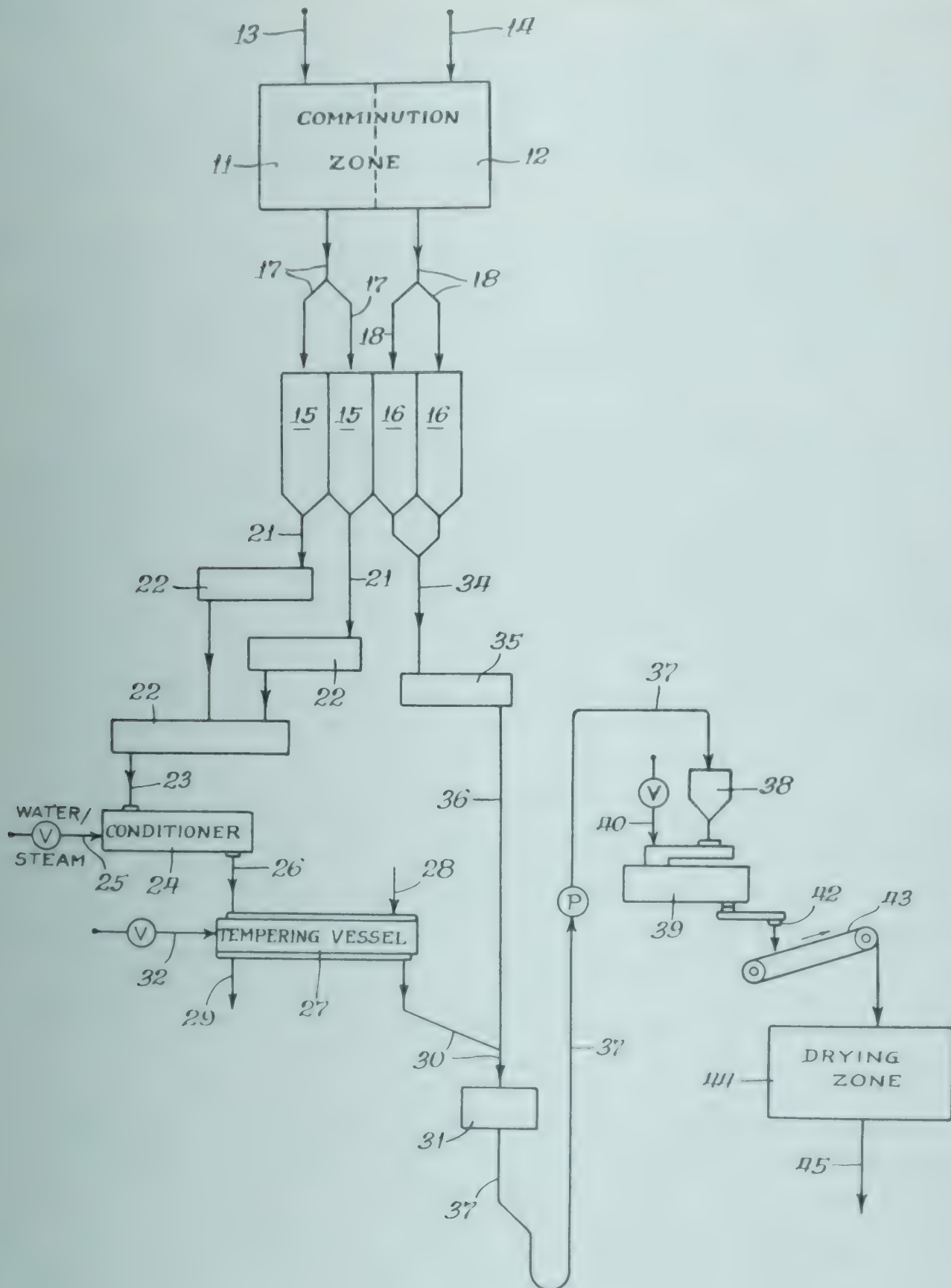
|                            | <u>Pounds</u> |
|----------------------------|---------------|
| Wheat middlings            | 10.00         |
| Corn gluten meal           | 10.00         |
| Dehydrated alfalfa         | 1.00          |
| Soybean meal               | 5.20          |
| Cottonseed meal            | 3.00          |
| Urea                       | 1.00          |
| Dicalcium phosphate        | 1.00          |
| Limestone                  | 0.25          |
| Fats                       | 1.00          |
| Mineral and vitamin premix | <u>0.55</u>   |
|                            | 100.00        |

The ground corn, ground milo, hominy feed and wheat middlings are introduced into the system (see Figure 11.2) through line 13 as the starch-bearing portion of the formulation. The remainder of the ingredients, i.e., generally nonstarch, are introduced through line 14. The introduction of the ingredients through each line is generally continuous in the proportions indicated in the above formulation. Thus, 77 pounds of the starch-bearing material are introduced per 23 pounds of the remaining ingredients or nonstarch component. The starch-bearing materials normally contain about 10% of water. In zone 24, an additional 12 pounds of water is added as live steam as 77 pounds of starch-bearing material to bring the starch-bearing material to a moisture content of about 22% (preferred range: 15 to 30% moisture).

At the time of water addition, sufficient heat is applied by an external heating source (not shown) to the conditioning zone 24 to bring the temperature of the mixture therein to about 180°F., the live steam assisting in raising and maintaining the temperature. The residence time in zone 24 is about 1 minute (conditioning zone range: 160 to 200°F. for 1 to 5 mins.). The mixture flows from zone 24 to zone 27 which is also maintained at 180°F. (tempering zone range: 160 to 200°F. for 10 to 60 mins.). The residence time in zone 27 is about 20 minutes and the resulting material, including swollen, water-permeated starch cells, after flowing through zone 27 in the 20 minute residence time, is mixed with the ration in mixer 31 and is then charged to the heated pressurized chamber of extruder 39.

Sufficient live steam is added to line 40 to bring the mixture to the 22% water level and heat is applied to the zone by heating means (not shown) in the extruder structure to attain a temperature of about 250°F. The additional water, e.g., 3.6 pounds per 112 pounds of total mixture, from line 40 is required for the nonstarch portion of the formula which entered the system at an approximate 10% water level. The pressure zone is maintained at a pressure of about 15 psig and the residence time of the material in the zone is about 2 minutes (pressure zone range: 1 to 50 psig for 1 to 5 mins.), whereafter the material is extruded to atmospheric pressure, dried at 44 and recovered at 45. The resulting material includes all of its starch cells in ruptured or exploded condition, releasing the food value therefrom.

FIGURE 11.2: PRETREATMENT OF STARCH COMPONENT OF MIXED FEEDSTUFF





## Digestible Feed for Baby Animals

R.A.S. Templeton (U.S. Patent 2,971,843; February 14, 1961) describes the preparation of a highly digestible animal feed especially suited for baby animals in the first weeks of life.

Example: Into a dry food mixer preferably of the horizontal paddle type, having a convenient mixing capacity of 1,000 lbs. dry, the following ingredients are fed, namely,

|                           | Pounds |
|---------------------------|--------|
| Maize meal                | 300    |
| Barley meal               | 200    |
| Wheat meal                | 200    |
| Fish solubles             | 50     |
| Extracted ground-nut meal | 50     |
| Molasses                  | 100    |
| Salt                      | 10     |
| Limestone flour           | 10     |
| Bone meal                 | 10     |

making a total of 930 lbs., of which it can be assumed that approximately one half of the weight of the molasses and the fish solubles is water, say 75 lbs.

With the paddle having been brought into operation to effect admixture of the ingredients, 1,250 lbs. of water are added so as to obtain a total water content well above the minimum requirement of 50% by weight and the mixing action is continued for a period of one hour. In order to facilitate the action of the natural enzymes the temperature of the contents of the mixer is raised to between 90°F. and 100°F. and held at that point during this period by the admission of steam to the mixer in any convenient manner. A certain small addition to the water content will take place due to the admission of the steam on account of its condensation with the mixture; however, this is unobjectionable.

The water mixed partially predigested porridge-like contents are transferred by pump or by gravity to the cooker. While the cooker is being agitated by the paddle, steam is admitted to the cooker and the temperature therein is thereby raised to 212°F. and held at that point for a period of thirty minutes. The admission of the steam produces some further condensation which is not objectionable having regard to the amount of water used.

During this cooking, starches are substantially gelatinized, and proteins converted (over 180°F.). At 212° enzymes are inactivated and bacterial sterilization obtained. The water mixed predigested and precooked product now in rather more viscous porridge-like form is passed by gravity or by pump to the hopper of a standard type film drier (sometimes described as a steam heated cylinder). In the drier, the rotating steam heated cylinder picks up films of the product from the hopper and as the cylinders revolve the water present in the film, or the greater part thereof, is evaporated leaving dry films which are removed from the cylinder by doctor knives in thin paper-like sheets having a residual moisture content of approximately 12% water. These sheets fall onto a standard type of conveyor preferably perforated on which they are cooled. After cooling the sheets are broken into flakes on a

breaker and are then ready for packing and delivery.

While on the conveyor heat susceptible vitamin rich oil and antibiotics may be applied to the paper-like sheets in any convenient manner.

## MILK PRODUCTION

### Enzyme Additives to Stimulate Milk Production

J.W. Brooks (U.S. Patent 3,250,622; May 10, 1966; assigned to Pabst Brewing Company) found that certain enzymes and combinations of enzymes stimulate milk production in milk producing animals. These enzymes are gumase and combinations of gumase with protease and amylase. Although this finding is applicable to milk producing animals generally, it is especially important with respect to dairy cattle. These are administered orally, by incorporating them into a feed additive. A preferred additive contains 3,405,000 PV units per pound of proteolytic enzyme, 5,675,000 DV units per pound of amylolytic enzyme and 13,620 gumase units per pound, intimately associated with a ground malt carrier in proportions corresponding to about 40% of total enzymes to 60% of the ground malt carrier. This additive is referred to hereinafter as Additive A.

Example 1 — Florida: This test involved 196 head of milking cows in a commercial dry lot dairy operation. The test was conducted over a 41 day period between August 26 and October 5, of the test year. Crossbred cows of Brown Swiss, Guernsey, Jersey and Holstein breeding were employed in the test. Additive A was added to a manufactured complete feed at a level to supply 3 g. of enzyme supplement per head per day.

Results of the test are shown in the following table:

|  | September             |                |           | April through August  |                |           |
|--|-----------------------|----------------|-----------|-----------------------|----------------|-----------|
|  | Two Years Before Test | Preceding Year | Test Year | Two Years Before Test | Preceding Year | Test Year |
| Average daily milk production per cow, pounds..... | 24.4                  | 25.9           | 27.0      | 24.4                  | 26.9           | 25.9      |
| Average daily feed per cow, pounds.....            |                       |                | 21.5      | 26.1                  | 25.1           | 22.4      |

The use of Additive A in the ration of these cows resulted in an improvement of 1.1 pounds of milk per head per day with a feed savings of 0.9 pound, when compared with the results of a 5 month period without Additive A in the ration. This is of particular significance when one considers that September normally is the poorest milk production month in the Florida area.

Example 2 — Canada: In this test, 10 pairs of cows from an outstanding Jersey herd were selected on the basis of calving dates and level of milk production. One cow in each pair was fed Additive A (3 grams per head per day) and the other a control ration for 54 days. The results are given in the table on the following page.



## Ruminant Feeds

| Group           | Average Daily Milk Production |                     | Drop in Milk Production, Percent | Average Daily Grain Intake, Pounds |
|-----------------|-------------------------------|---------------------|----------------------------------|------------------------------------|
|                 | 60-Day Pre-Test, Pounds       | 54-Day Test, Pounds |                                  |                                    |
| Control.....    | 31.5                          | 28.7                | 8.9                              | 12.2                               |
| Additive A..... | 30.8                          | 28.7                | 6.8                              | 11.6                               |

During the 54 day test the Additive A group held its milk production better than the controls on 0.6 pound less feed. This benefit over a 100 day period would result in 2.1% more milk and a 60 pound savings in feed per cow.

## UTILIZATION OF NONPROTEIN NITROGEN

Ruminant animals have the unique ability of utilizing nonprotein nitrogenous compounds. Ruminant animals which include cattle and sheep, and which chew their cud, have a complex stomach of several compartments. The first stomach, lying next to the reticulum, is known as the rumen. It is generally believed that in the rumen, nitrogen fed to the animal is converted into protein. Conventionally, the major portion of nitrogen required by the animal is supplied in the form of grain protein. However, in recent years there has been a trend toward supplying a portion of the nitrogen, perhaps as much as one-third, by incorporating urea in the feed. As a nonproteinaceous source of nitrogen, urea is attractive in feeding ruminants because it represents a more concentrated and economical form of nitrogen. Pure urea contains about 46.65% N, but the amount that may be safely incorporated in animal feeds is limited by the fact that it possesses a definite toxicity limit toward ruminants. These processes deal with practical and economical ways of preparing and utilizing urea feed supplements.

### Urea Addition Complexes

The process of H.W. Titus (U.S. Patent 3,180,735; April 27, 1965; assigned to Limestone Products Corporation of America) provides a safe readily assimilable form of urea for use as a feed supplement, in the form of addition complexes of unsubstituted urea. Addition complexes of unsubstituted urea with inorganic acids which may be employed include, for example, urea phosphate,  $\text{CO}(\text{NH}_2)_2 \cdot \text{H}_3\text{PO}_4$ , rhombic crystals, readily soluble in water. Addition complexes with inorganic salts include: Urea-sodium chloride-water,  $\text{CO}(\text{NH}_2)_2 \cdot \text{NaCl} \cdot \text{H}_2\text{O}$ , rhombic prisms, MP  $60^\circ$  to  $70^\circ\text{C}.$ , readily soluble in water. Urea-calcium sulfate,  $4\text{CO}(\text{NH}_2)_2 \cdot \text{CaSO}_4$ .

To make the most efficient use of the urea addition complexes, it is advantageous to employ two or more of the complexes in a given concentrate, the choice being determined by the desired additional inorganic elements, such as phosphorus, sulfur, calcium, and the like. The balance of feed supplement can comprise any suitable grain material, such as corn, oats, barley, wheat, milo, oilseed meal, in any suitable form, whole, flaked or ground. In the preparation of feed concentrates there may be included with the urea complex various inorganic ingredients such as calcium carbonate (limestone), vitamins, commercial trace element premixes, and so on.

## Ruminant Feeds

The ingredients are dry mixed, in comminuted form, in the proportions indicated.

### Example 1: Urea, Urea-Phosphoric Acid Complex Supplements

| Ingredient                        | Percent       |               |
|-----------------------------------|---------------|---------------|
|                                   | Typical       | Optimal range |
| Urea-phosphoric acid complex..... | 17.35         | 15-20         |
| Urea (free urea).....             | 28.35         | 25-30         |
| Barley.....                       | 54.30         | 50-60         |
| <b>Total.....</b>                 | <b>100.00</b> | <b>100</b>    |

### Example 2: Urea, Urea-Calcium Sulfate Complex Supplements

| Ingredient                        | Percent       |               |
|-----------------------------------|---------------|---------------|
|                                   | Typical       | Optimal range |
| Urea-calcium sulfate complex..... | 22.95         | 20-20         |
| Urea (free urea).....             | 28.35         | 25-30         |
| Wheat.....                        | 48.70         | 40-55         |
| <b>Total.....</b>                 | <b>100.00</b> | <b>100</b>    |

### Example 3: Urea-Mineral Concentrate

| Ingredient                            | Percent       |               |
|---------------------------------------|---------------|---------------|
|                                       | Typical       | Optimal range |
| Urea-phosphoric acid complex.....     | 17.00         | 15-20         |
| Urea-calcium sulfate complex.....     | 22.55         | 20-25         |
| Urea-sodium chloride-water complex..  | 19.50         | 15-20         |
| Urea.....                             | 27.90         | 15-30         |
| Calcium carbonate (or limestone)..... | 11.40         | 8.5-33        |
| Commercial trace-mineral pre-mix..... | 1.65          | 1.5-2         |
| <b>Total.....</b>                     | <b>100.00</b> | <b>100</b>    |

### Example 4: Urea-Mineral-Vitamin Concentrates

Urea-mineral concentrate above plus 12,000,000 to 144,000,000 I.U. of vitamin A per ton and 1,200,000 to 14,400,000 I.U. of vitamin D per ton.

### Example 5: Complete Feed Composition

| Ingredient:                            | Parts by weight  |
|--|------------------|
| Ground barley .....                    | 600              |
| Crimped oats .....                     | 600              |
| Wheat bran .....                       | 600              |
| Cane molasses .....                    | 200              |
| Urea-mineral-vitamin concentrate ..... | <sup>1</sup> 120 |
|  | <b>2,120</b>     |

<sup>1</sup> Supplies about 46% of the total protein-equivalent.



## Clay and Sodium Propionate

I. J. Belasco (U.S. Patent 2,965,488; December 20, 1960; assigned to E. I. du Pont de Nemours and Company) found that feed compositions containing natural protein, urea, cellulosic matter, other carbohydrates and minerals, and at least one member of the class consisting of water-soluble acetate and water-soluble propionate, are greatly improved by incorporating therein a further additive which consists of an insoluble, fine divided solid capable of absorbing ammonia resulting from proteolysis of proteinaceous material, hydrolysis of urea and degradation of aminoacids, peptides and other nitrogen containing materials in the paunch of ruminant animals.

The solid additive capable of absorbing ammonia is preferably a clay or clay-like material, including the various alumino silicates which are obtainable in nature in finely divided or even colloidal form. Suitable materials include attapulgites, kaolines, etc.; diatomaceous earths, hydrated silicas, cation-exchange resins, and other solid surfaces capable of retaining ammonia loosely, e.g. chemisorption. The weight ratio of this solid additive should be about 2% to 100%, preferably 5% to 50%, of the weight of urea.

The benefits of this process are observed when sheep and cattle are fed the mixtures herein disclosed. They can also be demonstrated by the use of the rumen technique which has been developed during recent years, whereby the changes taking place in feeds, brought about by microorganisms, can be measured more readily and accurately. This technique involves the use of an apparatus in which the digestive processes of the animal are conducted and can be studied in vitro. By this means, various animal feeds are introduced into or withdrawn from the laboratory unit under carefully controlled conditions and the changes taking place studied critically and progressively during the consumption of the feed by the microorganisms.

The benefits of employing propionate simultaneously with clay are shown in the tables below.

**Example 1: QUANTITIES USED**

|                              | 1     | 2                | 3     | 4     |
|------------------------------|-------|------------------|-------|-------|
| Cellulose, g.....            | 0.5   | 0.5              | 0.5   | 0.5   |
| Molasses ash, g.....         | 0.03  | 0.03             | 0.03  | 0.03  |
| Nutrient Salt soln., ml..... | 15    | 15               | 15    | 15    |
| Starch, g.....               | 0.1   | 0.1              | 0.1   | 0.1   |
| Urea, g.....                 | 0.080 | 0.080            | 0.080 | 0.080 |
| Sodium acetate, g.....       |       | 0.200            |       | 0.200 |
| Attapulgite, g.....          | 25    | 25               | 25    | 25    |
| Rumen inoculum, ml.....      |       | dilute to 50 ml. |       |       |
| H <sub>2</sub> O.....        |       |                  |       |       |

**Example 2: QUANTITIES USED**

|                              | 1     | 2                | 3     | 4     |
|------------------------------|-------|------------------|-------|-------|
| Cellulose, g.....            | 0.5   | 0.5              | 0.5   | 0.5   |
| Molasses ash, g.....         | 0.03  | 0.03             | 0.03  | 0.03  |
| Nutrient Salt soln., ml..... | 15    | 15               | 15    | 15    |
| Starch, g.....               | 0.1   | 0.1              | 0.1   | 0.1   |
| Urea, g.....                 | 0.080 | 0.080            | 0.080 | 0.080 |
| Sodium propionate.....       |       | 0.200            |       | 0.200 |
| Diatomaceous earth, g.....   | 25    | 25               | 25    | 25    |
| Rumen inoculum, ml.....      |       | dilute to 50 ml. |       |       |
| H <sub>2</sub> O.....        |       |                  |       |       |

**RESULTS OF TESTS**

|                                   | 1      | 2      | 3      | 4      |
|-----------------------------------|--------|--------|--------|--------|
| Residual ammonia, percent.....    | 0.0589 | 0.0584 | 0.0521 | 0.0467 |
| Urea utilization, percent.....    | 55.0   | 55.4   | 63.7   | 69.8   |
| Cellulose digestion, percent..... | 87.4   | 86.9   | 86.9   | 87.1   |

**RESULTS OF TESTS**

|                                   | 1      | 2      | 3      | 4      |
|-----------------------------------|--------|--------|--------|--------|
| Residual ammonia, percent.....    | 0.0607 | 0.0555 | 0.0551 | 0.0528 |
| Urea utilization, percent.....    | 48.5   | 54.8   | 54.9   | 63.5   |
| Cellulose digestion, percent..... | 79.7   | 81.4   | 79.0   | 78.7   |

## Ruminant Feeds

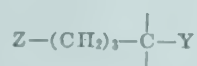
Example 3: A finished feed mixture, suitable for dairy cattle and feed-lot diets for ruminants is prepared by grinding and mixing the following.

|                                  | Parts by weight |
|----------------------------------|-----------------|
| Timothy hay .....                | 955             |
| Dehydrated alfalfa .....         | 42              |
| Yellow corn .....                | 600             |
| Crimped oats .....               | 300             |
| Soybean meal .....               | 30              |
| Urea (42% N) .....               | 40              |
| Calcium propionate .....         | 40              |
| Colloidal attapulgite clay ..... | 20              |
| Trace mineralized salt .....     | 40              |
| Dicalcium phosphate .....        | 30              |

### 3,4-Dihydro-2H-Pyran-2-Carboxylate Monohydrate

H.T. Peeler (U.S. Patent 3,336,136; August 15, 1967; assigned to International Minerals and Chemical Corporation) has a process to provide supplemented animal feedstuffs containing a relatively large amount of the protein equivalent as nonprotein nitrogen compounds and containing a chemical composition biologically equivalent to lysine (i.e., stimulating weight gain and feed efficiency).

The chemical compositions are compounds of the group consisting of 7-oxo-6,8-dioxabicyclo (3,2,1) octane; 3,4-dihydro-2H-pyran-2-carboxylic acid and the C<sub>7</sub>-C<sub>12</sub> esters, C<sub>6</sub>-C<sub>12</sub> amides, and nontoxic salts thereof; and compounds of the formula:



wherein Z is of the group consisting of primary and C<sub>1</sub> to C<sub>6</sub> amide radicals, primary amino radicals, aldehyde radicals, C<sub>3</sub> to C<sub>7</sub> 1,1-dialkoxy methyl radicals, carboxyl radicals, and C<sub>1</sub> to C<sub>6</sub> esters and nontoxic salts of carboxyl radicals; Y is of the group consisting of primary and C<sub>1</sub> to C<sub>6</sub> amide radicals, aldehyde radicals, C<sub>3</sub> to C<sub>7</sub> 1,1-dialkoxy methyl radicals, carboxyl radicals and C<sub>1</sub> to C<sub>6</sub> esters and nontoxic salts of carboxyl radicals; and the two unspecified carbon valences are satisfied by one of the group consisting of (1) a hydrogen and a primary amino radical, (2) a hydrogen and a hydroxyl radical, and (3) an oxo oxygen radical; and mixtures thereof. The ammonium, sodium, potassium, magnesium, calcium and zinc salts of the carboxylic acids are preferred. A specifically preferred salt is sodium 3,4-dihydro-2H-pyran-2-carboxylate monohydrate.

In general the chemical compositions are fed to the animal in an amount of from about 0.002% to about 2.0% by weight of the animal feed supplement. The lysine activity of the chemical compositions of this process is particularly effective when the animal diet contains urea or an ammonium salt as a protein source and is particularly effective when at least 5%, more preferably 10%, of the protein of the animal feed is provided as urea.

Example: Two calves in a carefully controlled environment are fed a supplemented ratio containing a high level of urea. The supplement added to the ratio contains 64% protein wherein half of the protein equivalent is made up by urea. One pound of the supplement is added to the daily ration for each animal. Substantially duplicate rations are fed to the



## Ruminant Feeds

calves except ten milligrams of sodium 3,4-dihydro-2H-pyran-2-carboxylate monohydrate per pound of supplement is added to the one pound of supplement mixed with the daily feed ration for one of the calves.

The feed intake and metabolism of the calves are carefully determined and a nitrogen balance is calculated. The calf fed the sodium carboxylate salt exhibits a greater weight gain, increased nitrogen retention and a higher feed efficiency.

The high-urea 64% supplement had the following composition:

| Ingredient:                                    | Lbs.         |
|--|--------------|
| Soybean meal (44%) -----                       | 503.2        |
| Alfalfa meal -----                             | 140.0        |
| Molasses, cane -----                           | 140.0        |
| Urea (262% protein equivalent) -----           | 145.8        |
| Dicalcium phosphate -----                      | 52.0         |
| Salt (NaCl) -----                              | 17.0         |
| Vitamin A and D concentrate <sup>1</sup> ----- | 2.0          |
|  | <hr/> 1000.0 |

<sup>1</sup> Stabilized dry vitamin A and D concentrate containing 4,540,000 USP units vitamin A and 567,500 USP units of vitamin D per pound.

### Coated Urea

L.I. Hansen (U.S. Patent 3,295,984; January 3, 1967; assigned to Archer-Daniel-Midland Company) discovered that animal feed supplements such as urea and other nonprotein nitrogen sources (e.g., ammonium phosphate) can be effectively fed to ruminants at considerably higher levels than previously expected, provided that the urea is first encapsulated with a cured coating of a curable copolymer of dicyclopentadiene and ester of unsaturated acid. By feeding this to ruminants, a number of advantages accrue. First, the encapsulating coating (which is water-insoluble) prevents the urea from hydrolyzing at too rapid a rate. Consequently, more urea is made available for efficient utilization by the host animal. Secondly, the deleterious side effects which accompany the use of uncoated urea are substantially avoided. Third, the palatability of the nonprotein nitrogen source is apparently improved. Fourth, the dietary needs of the host animal can be more effectively met under grazing conditions since more urea can be ingested by the ruminant based on the weight of roughage (i.e., cellulose) consumed by the animal.

In coating animal feed supplementals substantial benefits are obtained when a plurality of coatings (of one or more types) are employed, as opposed to a single coating. A single coating, regardless of amount, is generally inferior to a plurality of coatings of the same type, when the same total coating weight is employed. For ruminant feeding, the total weight of coatings (of all types) will usually be from 2 to 12%, more usually from 3 to 10%, e.g., 4 to 8%, based on the weight of the uncoated feed supplement (e.g., diammonium phosphate). The equipment used for coating the feed supplements was a horizontally mounted drum about 36 inches long and 16 inches in diameter. Means were provided to allow hot air to be passed axially through the drum. The air flow rate could be varied from about 100 to 500 cfm. Typically, the air flow rate was about 200 to 350 cfm. An open gas flame was used to preheat the air. Although the temperature of the air could be as high as 300°F. or higher, it was usually kept within the range of 140° to 220°F. During coating operations, the drum was rotated at a peripheral speed which could be varied from 50 inches per minute



to 500 inches per minute. Typically, the speed was about 130 to 140 inches per minute. Baffles located within the drum were used to cascade the granules and to reduce the tendency of the granules (or prills, etc.) to ball-up during the coating operation.

Example 1 — Preparation of Coated Feed Supplements: Granular, feed grade urea was coated with 3% of a dicyclopentadiene copolymer in the following manner. The uncoated urea was placed in the rotating drum. The coating process was begun without first allowing the granules to preheat. At about the same time, air preheated to 160° to 170°F. was passed through the drum. Each coating or layer was applied to the urea by introducing the necessary amount of copolymer through a long piece of aluminum tubing and spraying the tumbling area granules with a thin stream of the copolymer.

Two coatings of the copolymer were applied, each amounting to 1 1/2% based on the weight of the uncoated urea. A 15 minute interval was allowed between the two coatings so that the first coating could dry at least to the point of incipient gelation before the second coating was applied. The peripheral drum speed was approximately 130 to 140 inches per minute. During the coating process, the temperature of the granules rose to about 160°F. After the last (i.e., the second) coating of the copolymer had been applied, the heat was removed and the hot, coated granules were allowed to tumble (with air still coming through the drum) until the coated urea granules were tack-free. Then, the coated urea was removed from the drum and tested.

The six hour leach was 61.7% and the 24 hour leach was 92%. This is in strong contrast to uncoated urea which is substantially completely leached in a matter of only a few minutes. The coating material used in this example was a rapid drying solvent solution of a copolymer of (1) dicyclopentadiene and (2) a maleinized vegetable oil. This copolymeric resin is commercially available under the trademark Admerol 351. The copolymer contained about 30 to 40% dicyclopentadiene, with the remainder being the modified vegetable oil.

Example 2: Granular 16-48-0 diammonium phosphate, which had been screened to remove all particles over 10 mesh in size, was coated with 5% of a copolymer of dicyclopentadiene and an unsaturated ester. These granules were preheated to 220°F. before beginning the coating operation. This temperature was maintained during coating. The coatings were applied in the following manner. First, two coatings were applied, each amounting to 1 1/2%; second, two coatings were applied, each amounting to 1%. A 10 minute interval was allowed between each coating. The 24 hour leach for this product was 16%.

### Calcium Stearate in Pelleted Urea Feed

When urea is a component of compositions used to produce pelleted feeds, one of the problems that arises is the decrease in production rate of the pelleted feeds. J.B. Reynolds and L.E. Craig (U.S. Patent 3,249,441; May 3, 1966; assigned to Nipak, Inc.) have found that treating crystalline, granular urea with calcium stearate prior to its incorporation in the feed mix which goes into the pelleting mill has a profound effect in boosting the pelleting rates so that they are similar to those when using formulations that contain no urea.

Furthermore, the calcium stearate treatment acts to reduce the caking tendencies of pelleted feeds containing the urea. It is important that the calcium stearate be added to the pure



## Ruminant Feeds

crystalline, granular urea before the other conditioning agents are added, since addition of calcium stearate to "regular feed-grade urea" (urea + 4% kaolin clay and 5% rice bran) does not give as satisfactory results.

Furthermore, addition of calcium stearate at levels similar to those used on the urea itself, to a complete mixed feed just before pelleting, gives no substantial change in pelleting results. It is therefore very important that, to obtain the best results, the application of calcium stearate to the pure crystalline, granular urea should be followed by the addition of other conditioning agents such as kaolin clay and rice bran, and subsequently utilizing this calcium stearate-treated urea as a component of pelleted animal feeds and in particular, pelleted ruminant feeds.

Other stearates besides calcium stearate may be used providing they are not toxic. Salts of other fatty acids including oleic, linoleic, palmitic, myristic and lauric acids may also be used. When calcium stearate is used, the optimum level is in the range of from about 0.1% to about 0.25% by weight of urea. Quantities less than 0.1% are harder to handle because of the difficulty in obtaining uniform distribution of the calcium stearate throughout the mass of urea particles, while levels above 0.5% appear unnecessary and wasteful.

Example 1: A particular ruminant feed was prepared both from regular feed-grade urea and from calcium stearate-treated feed-grade urea containing calcium stearate at the level of about 0.1% by weight based on the urea. The formulation used was as follows:

| Ingredient:           | Parts |
|-----------------------|-------|
| Soybean meal -----    | 61    |
| Feather meal -----    | 8     |
| Wheat middlings ----- | 7     |
| Feed-grade urea ----- | 5.3   |
| Limestone -----       | 5     |
| Rock phosphate -----  | 3.5   |
| Molasses -----        | 3     |
| Cottonseed meal ----- | 3     |
| Animal fat -----      | 1     |
| Wheat bran -----      | 1     |
| Salt -----            | 1     |
| Fura Meal 40 -----    | 1     |
| Trace minerals -----  | 0.2   |
|                       | <hr/> |
|                       | 100.0 |

The feed was pelleted with a 75 hp. pellet mill wherein the die used was 2 1/2 inch thick and provided with 12/64 inch holes. Operating conditions were kept essentially identical for pelleting two lots of the feed that differed only in the type of feed urea used. With the regular feed-grade urea, the production rate was 1.78 tons of pelleted feed per hour. With the calcium stearate-treated feed-grade urea containing calcium stearate at the 0.1% level, the production rate was 2.62 tons per hour; this represents a 47% increase in production rate by use of the calcium stearate-treated feed-grade urea.

### Pelleted Feedstuffs Containing Urea

T.E. Freese (U.S. Patent 3,416,928; December 17, 1968; assigned to Allied Chemical Corporation) developed a process for the production of feedstuffs containing urea that have reduced caking tendency and bin set, which is accomplished by mixing a urea prill material

## Ruminant Feeds

which contains at least 90% by weight of prills within the size range that said prills will pass through a 12 mesh screen and are retained on a 30 mesh screen, at least 5% of the total prills being less than 20 mesh and at least 70% being greater than 20 mesh (U.S. Standard Screen Series) with other ruminant feed ingredients to form a free-flowing, nonsegregating mixture which is compressed into pellets by conventional means.

Example: A pelleted ruminant feedstuff was prepared. About 28 pounds of the 12 to 30 mesh prilled urea was mixed with about 372 pounds of a basal cattle feed mixture to provide about 7% urea and about 50% total protein equivalent. The total mixture contained the following:

| Component:                    | Pounds |
|-------------------------------|--------|
| Urea -----                    | 28     |
| Gluten feed -----             | 32     |
| Salt -----                    | 16     |
| Defluorinated phosphate ----- | 12     |
| Limestone -----               | 36     |
| Molasses -----                | 12     |
| Added water -----             | 4      |
| Standard middlings -----      | 4      |
| Linseed meal -----            | 4      |
| Cottonseed meal -----         | 124    |
| Soybean meal -----            | 128    |
|                               | 400    |

The batch was mixed in a vertical mixer and in a high speed blender. After mixing the batch, the mix was heated to about 50°C. with steam and pelleted with a California pellet machine using a 12/64 inch die. The pellets were cooled to ambient temperature in a horizontal conveying-screen cooler prior to storage. The finished pellets exiting the cooler contained 7% urea, 3% molasses, 50% protein equivalent, and about 9% moisture. The highest practical production rate was found to be 3,080 pounds per hour. The pelleted feed required a 22 pound weight to break down the pack when evaluated in a bin set storage test.

### Amino Acid Amides and Acid Salts

D.G. Crosby and H.E. Johnson (U.S. Patent 3,256,095; June 14, 1966; assigned to Union Carbide Corporation) found that the amides of the nutritional amino acids and the acid salts thereof can be effectively utilized to replace the corresponding amino acids for the nutrition of animals or can be used with standard dietary formulations as supplements to insure the adequate intake of amino acids. The amides of the nutritional amino acids and the acid salts of the amides of the nutritional amino acids can be produced economically and in large quantity at low cost—a fraction of that of the amino acids—which insures a ready availability. The amides of the nutritional amino acids and the acid salts thereof can be utilized as feed supplements with greater facility than can the corresponding amino acids, as they are generally much less toxic, more soluble, and frequently more effective biologically.

It should be noted however, that while the amides of the sulfur containing nutritional amino acids have been reported to require the presence of relatively high concentrations of fat to function properly and efficiently, the acid salts of these sulfur containing amino acid amides have not been found to require the presence of fats to function at optimum efficiency. It is therefore preferred, in all food compositions where the sulfur containing nutritional amino acids are required, to use the nontoxic acid salts of the amides of the sulfur containing



amino acids.

In those food compositions where sulfur-free nutritional amino acids are required, either the amide of the sulfur-free nutritional amino acid or the acid salt thereof may be used. The amides of the nutritional amino acids can be administered to animals directly or as a supplement in combination with food or feedstuffs. They can be administered as crystalline powders, pastes, pellets, capsules or the like, or in solution; they may be mixed with other substances such as antibiotics, vitamins, minerals, or drugs generally, when simultaneous administration is desired. The acid salts of the nutritional amino acid amides can be administered in a manner similar. Likewise, the amino acid amide salts can be administered as aqueous solutions as they are highly water-soluble. These acid salts can also be more easily blended with other dietary ingredients as aqueous solutions.

For these reasons, it is preferred to use the acid salts of the nutritional amino acid amides. While the amides of the nutritional amino acids and the nontoxic salts thereof can be administered both directly and as supplements, the nontoxic acid salts can be administered to mammals intravenously. This factor is of extreme importance when it is desired or necessary to avoid ingestion. When the acid salts are to be used in this manner, it is preferred to use the hydrochloride, phosphate, citrate, or acetate salts as they are normal constituents of the blood.

By any mode of administration, both the amides of the nutritional amino acids and the nontoxic acid salts thereof can be used singly or in any combination or mixture. Indispensable and dispensable amino acid amides and/or the salts thereof may be used in any combination, in nutritional, nontoxic amounts. Both the amides of the nutritional amino acids and the nontoxic acid salts thereof should be administered in a nutritional amount or as necessary to bring the total amino acid intake into balance. This amount will vary from species to species for the particular amide or acid salt to be administered. It will also vary with the nutritional adequacy of the food. Generally, a nutritional amount will be from one to three times the daily requirement of the species for the corresponding amino acid. The amino acid requirements of the various animal systems are well known in the art.

Example: If the normal daily leucine requirement of a pig is one-tenth mol, then up to one-tenth mol of leucinamide or leucinamide acid salt can be substituted therefor. When the required amino acid is an optical isomer such as L-leucine, then twice the mol equivalent amount of DL-leucinamide or a nontoxic acid salt of DL-leucinamide would be required for complete effectiveness. In such a case, the D-form would pass unaffected through the system as waste with no toxic effects. As has been stated above, the amino acid amides and the nontoxic acid salts thereof are less toxic than the corresponding amino acids and up to at least three times the mol equivalent amount may be used with safety.

## FEED FOR SWINE

### PREVENTION OF IRON DEFICIENCY ANEMIA IN BABY PIGS

Iron deficiency anemia is a common nutritional problem encountered in raising young pigs which is responsible for reduced weaning weights and, in extreme cases can result in loss of the pigs from secondary causes attributable to nutritional anemia. The problem arises since sow's milk, normally the sole source of nourishment during the critical stage of pig development from farrowing to about four weeks of age, is unable to supply the iron requirements of the young pigs.

Various means of supplementing the mineral intake of the suckling pigs have been proposed, however, up until now none of these have been entirely satisfactory. For example, one of the older techniques was to dig up clumps of sod and place them where the young pigs could eat them. Such a method of prevention is not acceptable to present swine husbandry practices because the iron content of the soil is usually unknown and feeding sod may constitute a hazard in the spread of internal parasites and infectious diseases.

#### Sweetened Iron Supplement

A preparation for preventing iron-deficiency anemia in baby pigs has been developed by M.C. Wilkening (U.S. Patent 3,332,778; July 25, 1967; assigned to Nebraska Consolidated Mills Company). In order for the oral preparation to be effective in preventing anemia the iron must be present in high amounts, at least about 6.5% by weight. A preferred source of iron is ferrous fumarate.

The baby pigs prefer the sow's milk to feeds. In order for the prestarter to be effective in preventing anemia it must be highly palatable. The carrier, therefore, must contain ingredients to make the preparation attractive to baby pigs. A sweetening agent in the preparation accomplishes this purpose when present in amounts sufficient to attract the baby pigs, for example, from about 10 to about 14% by weight of the preparation. The preparation of the process is fed ad libitum by merely placing the preparation in pans for consumption for the first 21 days of baby pig's life.

A swine ration, fed ad libitum on which baby pigs showed good weight gains, hematocrit and hemoglobin levels is as shown on the following page.



## Feed for Swine

| <u>Ingredient</u>               | <u>Percent</u> |
|---------------------------------|----------------|
| Yellow corn meal                | 25.0           |
| Sugar (sucrose)                 | 10.0           |
| Wheat middlings                 | 5.0            |
| Fish meal (65%)                 | 5.0            |
| Dried whey                      | 10.0           |
| Fat (60% vegetable, 40% animal) | 3.0            |
| Dried buttermilk                | 10.0           |
| Dried skim milk                 | 10.0           |
| Iron fumarate                   | 20.0           |
| Pig blend                       | 1.0            |
| Defluorinated phosphate (18%)   | 0.8            |
| Swine mineral mix               | 0.2            |
| Total                           | 100.0          |

The pig blend consists of the following ingredients:

|  | <u>Percent</u> |
|--|----------------|
| Vitamin A (30,000 I.U./g.)                   | 1.47           |
| Vitamin D-3 (15,000 I.U./g.)                 | 1.10           |
| Riboflavin (4 g./lb.)                        | 3.75           |
| Nop-Cap 64 (58.8 g./lb. of pantothenic acid) | 0.85           |
| 50% niacin (227 g./lb.)                      | 0.42           |
| Vitamin B-12 supplement (60 mg./lb.)         | 1.17           |
| 37 1/2% choline chloride (147.5 g./lb.)      | 25.42          |
| T.M.-50 (50 g. oxytetracycline/lb.)          | 2.58           |
| BHT (a preservative)                         | 0.62           |
| Pig Nectar (a sweetening agent)              | 5.00           |
| Dried corn fermentation solubles (a carrier) | 57.62          |
| Total  | 100.00         |

The swine mineral mix consists of the following elements:

|           | <u>Percent</u> |
|-----------|----------------|
| Manganese | 6.00           |
| Iron      | 2.00           |
| Copper    | 0.20           |
| Cobalt    | 0.02           |
| Iodine    | 0.12           |
| Zinc      | 2.50           |
| Calcium   | 24.00          |

Humus-Soft Phosphate-Colloidal Clay-Iron Supplement

P.J. Geerlings (U.S. Patent 2,926,085; February 23, 1960) found that if the mineral salts containing iron and copper for prevention of anemia are incorporated in a mixture of humus and soft phosphate with colloidal clay, tiny pigs will lick it up without hesitation of their own free will, in proportion to their need. It can be fed to pigs at the age of 2 to 3 days. The humus which is used may be defined as a brown or black soil containing decomposed vegetable or animal matter and which contains from 10% to 60% moisture. The soft phosphate with colloidal clay is produced as a by-product in the process of mining phosphate rock in the area around Dunnellson, Florida and is also sometimes referred to as "colloidal phosphate".

Trace minerals are added to a mixture of humus and soft phosphate with colloidal clay to such an extent that the iron content preferably ranges between 0.04% and 0.20%. Edible salts soluble in water, such as ferric ammonium citrate, ferrous carbonate, ferrous sulfate, and iron saccharate are suitable. Likewise, copper salts (copper sulfate) should be present to provide copper preferably from 0.003% to 0.012% based upon the weight of the entire composition.

A preferred composition will also contain bone meal in the amount of 2% to 10% and trace minerals other than copper and iron, the total quantity of such minerals totaling from 1/2% to 5%. Other trace minerals include manganese, cobalt, nickel, potassium, zinc and sodium, which are present in the form of soluble edible salts. The bone meal aids in the production of a free-flowing material and improves the appearance of the product as well as enriching the product in calcium and phosphorous. If desired, antibiotics and vitamins may be added, but these are not essential.

Iron-Sphagnum Moss Preparation

H.D. Hutchinson (U.S. Patent 3,428,457; February 18, 1969; assigned to Moorman Manufacturing Company) describes an oral iron-sphagnum moss preparation for control of iron deficiency anemia in suckling pigs. A specific embodiment of this process which proved to be unusually effective in treating iron deficiency anemia in suckling pigs had the following composition (this mixture, hereinafter referred to as Preparation A, was prepared by first mixing all of the dry ingredients and then adding the molasses):

| <u>Ingredient</u>                  | <u>Parts by Weight,<br/>Pounds</u> |
|------------------------------------|------------------------------------|
| Ferrous sulfate                    | 10.0                               |
| Trace mineral mixture <sup>1</sup> | 0.05                               |
| Sphagnum moss                      | 55.0                               |
| Cane sugar                         | 20.0                               |
| Molasses                           | 15.0                               |
| Total                              | 100.05                             |

<sup>1</sup> Furnishing Cu, Mg, Mn, Zn, Fe, Co and I.



## Feed for Swine

Preparation A is desirably light or bulky. For example, one handful will weigh approximately 3 oz. In testing the effectiveness of the above listed iron-sphagnum moss preparation, a series of five experiments were run. The pigs were started on the preparation from the time that they were three days of age until they were about five weeks old. The injections were usually given at the time they were three days old and supplied a level of 100 mg. iron per pig.

In the following tables when reference is made to feeding a certain amount of Preparation A (e.g., 2 oz. Prep. "A"/litter/day) this is intended to indicate that the stated amount of Preparation A is made available to the litter on a free choice basis. Ordinarily the stated amount will not be fully consumed by a litter even if it is a large one containing as many as 15 animals. However, the efficiency of the preparation is of such a high order that even a very minor amount thereof will supply the iron requirements of even large litters.

Summary of Hemoglobin and Gain Data from all Experiments in which the  
Iron-Sphagnum Moss Mixture (Preparation "A") Was Used

| Experiment Number | Treatment   | Hemoglobin (g./100 ml. blood) | 3-week Weight (lb.) | Weaning Weight (lb.) | Weaning Age (Days) |
|-------------------|---|-------------------------------|---------------------|----------------------|--------------------|
| I                 | {No dietary supplement or injection.....                              | 9.0                           | 12.2                | 30.0                 | 42                 |
|                   | {Armidxan <sup>1</sup> injection only.....                            | 11.5                          | 12.5                | 32.0                 | 42                 |
|                   | {Armidxan plus 2 oz. Prep. "A"/litter/day.....                        | 13.1                          | 12.1                | 32.3                 | 42                 |
|                   | {2 oz. Prep. "A"/litter/day.....                                      | 12.2                          | 12.4                | 31.7                 | 42                 |
| II                | {No dietary supplement or injection.....                              | 8.8                           | 12.2                | 22.0                 | 36                 |
|                   | {Armidxan injection.....  | 11.7                          | 13.2                | 24.1                 | 36                 |
|                   | {Armidxan plus 2 oz. Prep. "A"/litter/day.....                        | 12.4                          | 12.4                | 22.1                 | 36                 |
|                   | {2 oz. Prep. "A"/litter/day.....                                      | 12.2                          | 12.5                | 22.4                 | 36                 |
|                   | {Armidxan plus 6 oz. Prep. "A"/litter twice weekly.....               | 12.7                          | 13.0                | 22.6                 | 36                 |
|                   | {6 oz. Prep. "A"/litter twice weekly.....                             | 12.6                          | 13.3                | 23.7                 | 36                 |
| III               | {No dietary supplement or injection.....                              | 5.9                           | (No weights taken)  |                      |                    |
|                   | {Armidxan injection only.....   | 7.8                           |                     |                      |                    |
|                   | {2 oz. Prep. "A"/litter/day.....                                      | 10.9                          |                     |                      |                    |
| IV                | {No dietary supplement or injection.....                              | 9.3                           | 10.9                | 16.2                 | 34                 |
|                   | {Armidxan injection only.....   | 10.9                          | 11.5                | 17.6                 | 34                 |
|                   | {Armidxan plus 2 oz. Prep. "A"/litter/day.....                        | 11.9                          | 11.4                | 19.6                 | 34                 |
|                   | {2 oz. Prep. "A"/litter/day.....                                      | 11.4                          | 11.4                | 19.1                 | 34                 |
|                   | {Armidxan plus 6 oz. Prep. "A"/litter twice weekly in Creep Area..... | 12.0                          | 11.0                | 18.0                 | 35                 |
|                   | {6 oz. Prep. "A"/litter/twice weekly in Creep Area.....               | 12.2                          | 10.6                | 16.5                 | 35                 |
|                   | {Armidxan plus 6 oz. Prep. "A"/litter twice weekly in sow pen.....    | 12.6                          | 11.7                | 18.8                 | 35                 |
|                   | {6 oz. Prep. "A"/litter twice weekly in sow pen.....                  | 12.1                          | 10.7                | 17.9                 | 35                 |
| V                 | {No dietary supplement or injection.....                              | 7.7                           | 11.8                | 19.4                 | 34                 |
|                   | {Armidxan injection only.....   | 10.8                          | 12.7                | 21.5                 | 34                 |
|                   | {6 oz. Prep. "A" (0.5% Fe)/litter twice weekly.....                   | 10.2                          | 12.8                | 21.2                 | 34                 |
|                   | {6 oz. Prep. "A" (2.0% Fe)/litter twice weekly.....                   | 11.3                          | 11.5                | 19.8                 | 34                 |
|                   | {6 oz. Prep. "A" (4.0% Fe)/litter twice weekly.....                   | 10.5                          | 12.4                | 20.3                 | 34                 |
|                   |   |                               |                     |                      |                    |

<sup>1</sup> Armidxan is an iron-dextran complex marketed by Armour Veterinary Laboratories, Kankakee, Illinois.  
NOTE.—Brackets indicate pigs in treatments were littermates.

### Feeding Sow Iron Agent

C.E. Barnhart and C.H. Chaney (U.S. Patent 3,259,500; July 5, 1966; assigned to The Kentucky Research Foundation) discovered that when the sow is fed a ration enriched in certain specified iron compounds and in comparatively massive amounts, for about two weeks prior to farrowing and for at least two weeks immediately subsequent to farrowing, the iron content in its milk available for building the pig's blood remains at a comparatively high

level, and that no supplementary source of iron for the pigs is necessary, no development of rickets takes place in the pigs, and no adverse effects are suffered by the sow. The same phenomenon is observed when the iron-enriched ration is fed to a sow beginning at farrowing and continuing for three weeks after farrowing. This is illustrated with reference to Table 1 and Table 2.

TABLE 1: IRON AGENTS FED BROOD SOWS STARTING AT FARROWING

| Sow Ration  | Number Sows | Number Pigs | Pigs at 15 days |                | Pigs at 21 days |                |
|---|-------------|-------------|-----------------|----------------|-----------------|----------------|
|   |             |             | Hemoglobin      | Hematocrit     | Hemoglobin      | Hematocrit     |
|   |             |             |                 | <i>Percent</i> |                 | <i>Percent</i> |
| Basal (no iron)   | 8           | 64          | 6.3             | 26             | 6.0             | 25             |
| Basal plus 600 mg. Fe/lb. total ration ferrous fumarate           | 21          | 160         | 8.1             | 33             | 8.8             | 35             |
| Basal plus 900 mg. Fe/lb. total ration ferrous fumarate           | 4           | 33          | 9.2             | 37             | 9.3             | 35             |
| Basal plus 300 mg. Fe/lb. total ration ferrous fumarate           | 4           | 32          | 7.4             | 31             | 7.7             | 30             |
| Basal plus 600 mg. Fe/lb. total ration ferric citrate             | 2           | 16          | 8.3             | 34             | 8.4             | 33             |
| Basal plus 600 mg. Fe/lb. total ration ferric phosphate (soluble) | 2           | 16          | 9.1             | 36             | 9.5             | 36             |
| Basal plus 600 mg. Fe/lb. total ration ferric tartrate            | 2           | 22          | 8.8             | 35             | 8.7             | 35             |

TABLE 2: IRON AGENTS FED BROOD SOWS FOR TWO WEEKS BEFORE FARROWING AND FOR THREE WEEKS THEREAFTER

| Sow Ration  | Number Sows | Number Pigs | Pigs at 15 days |                | Pigs at 21 days |                |
|---|-------------|-------------|-----------------|----------------|-----------------|----------------|
|   |             |             | Hemoglobin      | Hematocrit     | Hemoglobin      | Hematocrit     |
|   |             |             |                 | <i>Percent</i> |                 | <i>Percent</i> |
| Basal (no iron)   | 11          | 88          | 6.2             | 23             | 5.7             | 23             |
| Basal plus 600 mg. Fe/lb. total ration ferrous fumarate           | 8           | 68          | 8.6             | 35             | 8.9             | 36             |
| Basal plus 600 mg. Fe/lb. total ration ferric citrate             | 2           | 12          | 8.1             | 32             | 9.0             | 33             |
| Basal plus 600 mg. Fe/lb. total ration ferric phosphate (soluble) | 2           | 18          | 8.4             | 34             | 9.9             | 38             |

It is desirable that the iron-enriched ration fed as a swine feedstuff have its iron-containing agent dispersed substantially homogeneously with the remainder of the ration. This preferably is accomplished by first providing a uniform concentrate or premix including all of that iron-containing agent and thereafter mixing that concentrate or premix homogeneously with the larger bulk of the total ration thus diluting the iron content of the premix to the ratios herein disclosed. The specific gravity of the iron-containing agent is appreciably greater than that of any other ingredient of the normal ration and care must be exercised to guard against a later separation of the iron-containing agent from the remainder of the premix prior to preparation of the total ration.



## PROMOTING LEANNESS OF CARCASS IN SWINE

Attempts are being made continuously to increase the in-carcass protein to fat ratio and the feed efficiency of pigs. Limited success has been achieved in this regard by increasing protein content in pig feed and/or by restricting the amount of pig feed given daily. However, it has been found that increasing the protein content of feed increases the cost of the feed and thereby increases the cost of production of bacon and also still results in a carcass which is quite high in fat content. Restricting the feed intake of pigs increases the time required for the pigs to reach a marketable weight and also either requires expensive feed metering devices or extra labor with its attendant increase in cost.

These processes are concerned with increased leanness in carcass, that is an increased protein to fat ratio achieved by including additives to basal feed without an attendant increase in production costs.

### Nitroalcohols and Nitrocarbamates

M.C. Bachman, J.L. Martin and J.M. Pensack (U.S. Patent 2,924,523; February 9, 1960; assigned to Commercial Solvents Corporation) discovered that swine fed nitroalcohols and nitrocarbamates are lean and show a decided decrease in the amount of back fat formed during growth, while maintaining essentially the same degree of feed utilization and rate of growth.

Example: The efficacy of this process was demonstrated by feeding pigs complete feeds containing 100 g./ton of 2-nitro-2-methyl-1-propanol and 2-nitro-2-propyl-1,3-propanediol respectively and comparing the depth of back fat on these pigs with the back fat of pigs fed the same complete feed containing no additives, duplicate groups of pigs being fed in each case. Different feed formulas were fed to the pigs at various times during their growth. Thus, a high protein content feed was fed to the younger pigs, while a high carbohydrate content feed was fed to the pigs during the final stages of growth when the pigs are normally fattened for market.

The amount of back fat formed on the pigs was determined by taking measurements of the fat depth at three points on the backs of the pigs when they weighed about 150 and 200 lbs. The fat depth was measured by means of a Duncan Lean Meter. This instrument uses the electrical conductivity differential between fat and muscle to indicate whether the measuring needle is embedded in adipose tissue or in muscle. The measured depth of needle penetration, less skin thickness, determines the thickness of fatty deposits. The following table sets out the relative amounts of back fat when pigs fed the above feeds weighed approximately 150 and 200 pounds.

DEPTH OF BACK FAT AT 150 LBS.

| Complete Feed                      | No. Pigs | Av. Wt. (Lbs.) | Back-Fat (Inches)         | Percent Response |
|------------------------------------|----------|----------------|---------------------------|------------------|
| Control-----                       | 19       | 149.5          | 1.534-1.262-1.105=1.300-- | Leaner 2.6%.     |
| Control+ Additive 1 <sup>1</sup> . | 20       | 152.5          | 1.444-1.239-1.115=1.266-- |                  |
| Control+ Additive 2 <sup>2</sup> . | 18       | 151.3          | 1.317-1.178-1.033=1.176-- | Leaner 9.5%.     |

(continued)

(continued)

DEPTH OF BACK FAT AT 200 LBS.

|                                       |    |       |                           |                 |
|---------------------------------------|----|-------|---------------------------|-----------------|
| Control.....                          | 19 | 202.4 | 1.884—1.660—1.385=1.643.. |                 |
| Control+<br>Additive 1 <sup>1</sup> . | 20 | 201.0 | 1.763—1.523—1.293=1.526.. | Leaner<br>7.1%. |
| Control+<br>Additive 2 <sup>2</sup> . | 18 | 200.9 | 1.775—1.580—1.283=1.546.. | Leaner<br>5.9%. |

<sup>1</sup> 2-nitro-2-methyl-1-propanol.<sup>2</sup> 2-nitro-2-propyl-1,3-propanediol.

### Ditnitro Compounds

Similarly, J.L. Martin (U.S. Patent 2,924,524; February 9, 1960; assigned to Commercial Solvents Corporation) found a 7 to 8% decrease in the depth of back fat formed during growth when swine feed contained dinitro compounds such as 2,2-dinitro-1,3-propanediol, and 2,2-dinitro-1-propanol in amounts of 75 g. per ton of feed.

### Nicotine

H.M. Cunningham (U.S. Patent 3,252,802; May 24, 1966; assigned to Canadian Patents and Development Limited) discovered that the use of nicotine or an ingestible salt of nicotine as an animal feed supplement will increase the ratio of protein to fat deposited in the carcass of growing pigs and in many instances will at the same time increase feed efficiency. Nicotine sulfate having been found particularly useful because of its ready solubility in water, is commercially available as "Black-Leaf 40" which is a preparation containing 40% nicotine sulfate.

Example: This example was carried out to determine the effect of the oral administration of nicotine sulfate on growth weight, feed efficiency and carcass composition of pigs under conditions of ad libitum feeding. Twenty barrows and twenty gilts averaging 24 kg. in body weight were removed in lots of four from litters that contained either four males or four females within a weight range of 4 kg. (The control, basal, pig feed ration contained 16.49% protein.)

Each pig was marketed when its body weight, determined weekly, exceeded 87 kg. All pigs were fed the control ration containing no nicotine for 24 hours before slaughter in order to ensure a safe nicotine level in the carcass to permit sale to a packer. Chilled carcasses were graded and scored according to standard procedures (1959, National Bacon Hog Policy, Record of Performance in Advanced Registry for Pure Bred Pigs, Production Service, Canadian Department of Agriculture, Ottawa, Canada). One-half of each carcass (packer style) was frozen, weighed and finely minced in a machine (Cunningham et al, Canadian Journal of Animal Science, 41: 158, 1961).

The minced tissue from each pig was placed in a plastic bag, allowed to thaw at room temperature, thoroughly mixed in a large bowl-type Hobart mixer and sampled. Samples were analyzed for dry matter, nitrogen and ash by standard procedures. Fat was determined by a modification of the Saxon procedure for feces (Hawk et al, 1947) in which 2 g. samples were shaken before extraction with 8 ml. of a solution containing 5 volumes of water to 3 of concentrated hydrochloric acid (HCl). The results are shown on the following page.



## Feed for Swine

### Effects of Graded Levels of Nicotine on Average Growth Rate, Feed Efficiency and Carcass Composition of Growing Pigs

|  | Level of nicotine in the ration <sup>a</sup><br>(mg./kg.) |         |         |         | Standard<br>error |
|--|---|---------|---------|---------|-------------------|
|  | 0   | 5       | 10      | 20      |                   |
| No of pigs.....                                      | 10  | 10      | 10      | 10      | -----             |
| Initial body weight, kg..                            | 23.4  | 23.4    | 23.6    | 23.2    | -----             |
| Days on test.....                                    | 84.0  | 86.8    | 84.0    | 84.7    | -----             |
| Daily gain, gm.....                                  | 779   | 743     | 775     | 786     | ±19               |
| Carcass weight, kg.....                              | 68.1  | 69.1    | 68.6    | 70.4    | ±.53              |
| Dressing percentage.....                             | 77.4  | *78.7   | *78.3   | *78.6   | ±.51              |
| Wt. gain per kg. feed,<br>gm.....                    | 284   | 291     | 290     | 288     | ±7                |
| Carcass gain per kg.<br>feed, gm. <sup>b</sup> ..... | 222   | 234     | 231     | 230     | ±5                |
| Depth of back and loin<br>fat, in.....               | 1.57  | *1.50   | 1.53    | 1.59    | ±.03              |
| Loin eye area, in.....                               | 3.63  | 3.74    | 3.57    | *3.84   | ±.08              |
| Carcass length, in.....                              | 30.04   | *31.1   | *30.8   | *30.8   | ±.11              |
| Carcass score.....                                   | 62.3  | *70.6   | 67.4    | 63.6    | ±2.8              |
| Carcass grade, A, B, C,<br>resp.....                 | 6, 4, 0   | 7, 3, 0 | 4, 5, 1 | 4, 6, 0 | -----             |
| Carcass analyses:                                    |   |         |         |         |                   |
| Dry matter.....                                      | 57.17   | *54.08  | 55.86   | 55.54   | ±.60              |
| Protein.....   | 14.69   | *15.47  | *15.36  | *15.37  | ±.17              |
| Fat <sup>c</sup> .....                               | 39.23   | †35.26  | *37.36  | †36.80  | ±.74              |
| Ash.....   | 3.25  | 3.34    | 3.34    | 3.36    | ±.06              |

<sup>a</sup> Levels of 0, 5, 10 and 20 mg. per kg. of feed in this experiment were approximately equivalent to 0, .33, .67, and 1.33 mg. per kg. of body weight per day.

<sup>b</sup> Calculated by assuming an initial dressing percentage of 75% in weanling pigs at the beginning of the experiment.

<sup>c</sup> Fat was determined "by difference."

\*P < .05 using the Student-Newman, Keul method (Federer, 1955).  
†P < .01.

These results show that significant improvements in carcass quality were obtained at all levels of nicotine sulfate supplementation with the greatest differences apparent at the lowest level. This level (5 mg. per kg. of feed) resulted in an average decrease of 10% in the fat content of the carcasses and a 5.3% increase in the protein content. There were also significant improvements in dressing percentage, area of loin, depth of back fat, carcass length, and ROP (record of performance) carcass score. With respect to depth of back fat it should be noted that the figures in the table are significant if carcass weight is considered.

Rate of gain and feed efficiency were not significantly influenced by the level of nicotine sulfate supplementation but there was greater feed efficiency in all groups fed nicotine sulfate with a maximum increase of 5.4% in the carcass gain: feed level at the lowest level of nicotine sulfate. The pigs in group four not only had a significantly higher dressing percentage than the controls but their average market weight was 0.9 kg. greater. This resulted in carcasses which averaged 2.3 kg. heavier than those of the controls allowing them to have a slightly greater depth of back fat and yet a significantly lower percentage of chemically determined carcass fat. It is also believed probable that fat on other parts of the carcass was reduced to give the significant differences in carcass fat.

### Hypoglycemic Sulfonylurea

The process of J.A. Aeschlimann and B. Tabenkin (U.S. Patent 2,941,884; June 21, 1960; assigned to Hoffmann-La Roche Inc.) provides a feed composition containing hypoglycemic sulfonylurea. By hypoglycemic sulfonylurea is meant a compound containing a sulfonylurea linkage as a constituent part of its molecular structure and capable, after ingestion by the

animal, of causing a decrease in the content of glucose in the blood. It has been found that swine grown on animal feed compositions comprising conventional rations and, in addition, a hypoglycemic compound consume feed with greater efficiency. Additionally, it has been observed that swine grown on hypoglycemic sulfonylurea feed compositions, when butchered, have leaner carcasses than when grown on conventional feed compositions, and the carcasses contain a greater percentage of primal cuts.

Example 1: (A) A basal ration for pigs was made up by mixing the following ingredients in a batch feed mixer.

|  | <u>Weight (lbs.)</u> |
|--|----------------------|
| Corn   | 325.0                |
| Soybean oil meal   | 60.5                 |
| Meat scraps  | 22.0                 |
| Alfalfa hay  | 25.0                 |
| Salt   | 2.5                  |
| Antibiotic feed supplement (containing<br>per pound 3.6 g. of chlortetracycline)   | 2.5                  |
| Glucose  | 60.5                 |
| Dicalcium phosphate  | 2.0                  |
| B vitamin feed supplement (containing<br>per pound 2 g. of riboflavin, 4 g. of<br>pantothenic acid, 9 g. of nacin, 10 g.<br>of choline chloride and 60 mg. of<br>folic acid) | 0.25                 |
| Dry vitamin A (containing per pound<br>9,080,000 U.S.P. vitamin A units)   | 0.1                  |
| D-activated animal sterol (containing<br>per pound 1,360,000 U.S.P. vitamin<br>D units)  | 1.5                  |
| Total (approx. 14% protein)  | 501.85               |

(B) A hypoglycemic sulfonylurea ration for pigs was made up by mixing together, in a uniform blend, 25 g. of 1-n-butyl-3-p-tolylsulfonylurea together with all of the ingredients listed in Example 1(A) above, in the quantities there listed.

(C) A second hypoglycemic sulfonylurea swine feed was made by blending together uniformly 25 g. of 1-cyclohexyl-3-p-tolylsulfonylurea and all of the ingredients listed in Example 1(A) above, in the quantities there listed.

Example 2: Seventeen pigs weighing about 100 pounds each were divided into three groups, the first group containing five pigs, and the second and third groups each containing six pigs. The pigs in group 1 were fed on the ration described in Example 1(A). Those in group 2 were fed on the ration described in Example 1(B). Those in group 3 were fed on the ration described in Example 1(C). The pigs were slaughtered as they reached market weight, approximately 200 pounds. The table on the following page lists the significant observations.



## Feed for Swine

|  | <u>Group 1</u> | <u>Group 2</u> | <u>Group 3</u> |
|--|----------------|----------------|----------------|
| No. pigs                                     | 5              | 6              | 6              |
| Slaughter weight (lbs.)                      | 205.6          | 203.7          | 209.2          |
| Daily gain (lbs.)                            | 1.53           | 1.56           | 1.53           |
| Feed per cwt. gain (lbs.)                    | 401.4          | 353.3          | 379.4          |
| Warm dressing percent                        | 82.78          | 81.60          | 81.60          |
| Carcass length (cm.)                         | 73.8           | 74.0           | 75.4           |
| Leg length (cm.)                             | 50.9           | 51.5           | 51.7           |
| Ham length (cm.)                             | 34.7           | 35.4           | 35.5           |
| Carcass depth (cm.)                          | 35.7           | 35.3           | 35.8           |
| Carcass thickness (cm.)                      | 27.4           | 27.0           | 27.3           |
| Mean backfat thickness (in.)                 | 1.58           | 1.41           | 1.49           |
| Percent primal cuts of live wt. <sup>1</sup> | 47.47          | 48.48          | 47.95          |
| Area of loin eye (sq. in.)                   | 4.26           | 3.88           | 3.94           |

<sup>1</sup> Primal cuts — ham, loin, Boston butt, picnic, and bacon side.

## OTHER

### Dry Feed for Weaning Pigs

In swine husbandry it has been the usual practice to permit small pigs to obtain nourishment from the sow until they attain 8 or 9 weeks of age, at which time they are weaned. P.J. Geerlings (U.S. Patent 2,926,084; February 23, 1960) has developed a dry feed which is a complete substitute for sow's milk from the time the pigs are about two weeks old. An example of such a feed is as follows.

Example 1 — Preparation: The ingredients listed are mixed in any order to form a dry homogeneous mixture.

| <u>Ingredient</u>                                      | <u>Parts by Weight</u> |
|--|------------------------|
| Cereal   | 1100                   |
| Oil meal   | 100                    |
| Fish meal  | 100                    |
| Dried milk   | 400                    |
| Sugar  | 110                    |
| Bonemeal, limestone or other calcium salts             | 64                     |
| Vitamin A and D (dry)                                  | 10.8                   |
| Trace minerals   | 3                      |
| Yeast (dried)  | 60                     |
| Arsanilic acid   | 0.2                    |
| Aureomycin compound (0.4% aureomycin in inert carrier) | 50                     |

## Feed for Swine

The combination of yeast and arsanilic acid, especially when present in the feed with the large proportion of antibiotic, effectively destroys disease-producing bacteria and promotes the growth of healthy bacteria. The yeast may be of the moist variety.

Example 2 — Use: The dry meal thus produced was creep-fed to a group of pigs beginning at the age of 10 days to supplement the milk they were getting from the sow. At 3 weeks the pigs averaged 10 pounds each. They were separated from the sow and fed this dry meal exclusively for a period of ten days. At the end of 31 days the concentration of the meal was reduced by mixing 2 parts thereof with 5 parts of a mixture of less expensive prepared pig meal, ground rolled oats and ground corn, this mixture being fed to the pigs for the next 21 days. After the 52-day period, the weaning meal was discontinued completely and the diet was changed to a mixture of a standard pig meal and corn, fed free choice in separate feeders. At the end of 8 weeks, the normal weaning time, the pigs averaged 40 pounds each in weight.

A second group of pigs taken from the same herd were raised under identical conditions except with respect to the manner of feeding. This group was left with the sow for the usual 8 weeks, their diet being supplemented by prepared pig meals which contain all of the ingredients included in the meal of the process except the increased quantity of aureomycin and the combination yeast and arsanilic acid. At the end of 8 weeks these pigs weighed 25 pounds each, which is considered the national average.

A careful account was kept of the cost involved to feed both groups of pigs. This cost data indicated that pigs fed on the weaning meal and in accordance with the process cost 50% less per pound of pork produced than pigs weaned at the usual 8 weeks and fed in the usual manner on sow's milk supplemented with pig meal. The additional feed required for the sow while suckling the pigs, and the loss in weight of the sow during the suckling period were considered in arriving at the 50% figure.

### Mercaptoimidazole Compounds as Growth Promoters

A method of raising swine for meat production which can be applied with particular advantage to gilts and barrows has been developed by W. Burroughs, V.C. Speer and V.W. Hays (U.S. Patent 3,256,094; June 14, 1966; assigned to Iowa State University Research Foundation, Inc.). A 2-mercaptoimidazole compound is orally administered to the swine at a rate from 60 to 100 milligrams of the 2-mercaptoimidazole compound per 100 pounds of body weight per 24 hours.

In order to obtain the desired growth promotant response from the 2-mercaptoimidazole compound, it has been found to be essential to control the quantity of vitamin A in the diet to just meet minimum nutritional requirements (200 to 800 I.U.). A thyroxine-active substance is administered in conjunction with the 2-mercaptoimidazole compound. (0.0005 to 0.0025 milligram of thyroxine activity can be employed per milligram of the 2-mercaptoimidazole compound.)

Example: Castrated males (barrows) and females (gilts) weighing approximately 150 pounds per pig and averaging 5 months of age were self-fed the No. 2 ration listed on the following page, containing 20 milligrams of 1-methyl-2-mercaptoimidazole per pound of ration.



## Feed for Swine

### Swine Rations

| <u>Ingredient</u>         | <u>No. 1</u> | <u>No. 2</u> | <u>No. 3</u> |
|---------------------------|--------------|--------------|--------------|
| Ground yellow corn        | 1,638        | 1,626        | 1,614        |
| 50% soybean oil meal      | 270          | 270          | 270          |
| Limestone                 | 16           | 16           | 16           |
| Dicalcium phosphate       | 24           | 24           | 24           |
| Sodium chloride (salt)    | 10           | 10           | 10           |
| Trace mineral premix      | 2            | 2            | 2            |
| Vitamin-antibiotic premix | 40           | 40           | 40           |
| Tapazole premix           | None         | 12           | 12           |
| Protamone premix          | <u>None</u>  | <u>None</u>  | <u>12</u>    |
| Total (lbs.)              | 2,000        | 2,000        | 2,000        |

The Tapazole premix contained 160 grams of Tapazole powder (1-methyl-2-mercaptoimidazole) mixed with 47.6 lbs. of ground corn. The Protamone premix contained 14 grams of Protamone (1% thyroxine iodinated casein) mixed with 21.0 lbs. of ground corn. The vitamin-antibiotic premix contained 5.0 grams of vitamin D<sub>2</sub> (142,000 I.U./g.), 0.8 lbs. Merck vitamin mixture No. 84, 2.0 lbs. Aurofac 10, 0.5 lbs. Merck vitamin B<sub>12</sub> mixture (20 mg./lb.) and 36.5 lbs. of ground yellow corn.

### Performance of Pigs on Different Rations

| <u>21-Day Results</u>       | <u>No. 1</u> | <u>No. 2</u> | <u>No. 3</u> |
|-----------------------------|--------------|--------------|--------------|
| Initial liveweight (lbs.)   | 150          | 148          | 151          |
| Final liveweight (lbs.)     | 190          | 196          | 202          |
| Average daily gain (lbs.)   | 1.90         | 2.29         | 2.42         |
| Average daily ration (lbs.) | 7.30         | 7.38         | 7.66         |
| Feed/pound gain (lbs.)      | 3.84         | 3.22         | 3.17         |
| Backfat probe (inches)      | 1.61         | 1.46         | 1.40         |

## PET AND OTHER FEEDS

### PET FOOD

Food products especially designed for household pets such as dogs, cats and other domesticated mammals kept for pleasure rather than for utility represent a substantial segment of what is generally referred to as the pet food industry. Foods designed for such pets generally are made up of meat and meat by-products, fish and fish by-products, cereals and other nutritional ingredients in the form of wet, dry or semidry products. Such foods contain proteins and carbohydrates in substantial amounts and are marketed in cans, bags or flexible containers.

Acceptability of such product to both the pet and the pet owner are very important factors in the marketing of pet foods and it is important that any product designed for pets be acceptable to the owner, as well as appetizing and flavorful to the pet.

### Hydratable Food Forming Own Gravy

A hydratable animal food particularly characterized by its ability to form a gravy-containing mixture on addition of liquid may be prepared by the process of V.D. Ludington, R.E. Schara and R.E. Mohile (U.S. Patent 3,119,691; January 28, 1964; assigned to General Foods Corporation).

Example: In accordance with a specific embodiment of this process a mixture of the following components was employed as charge.

|                       |      |
|-----------------------|------|
| Hominy feed           | 37 % |
| Wheat grey shorts     | 13 % |
| Corn germ meal        | 9.6% |
| Solvent soya          | 16 % |
| 52% meat meal         | 17 % |
| Wheat germ meal       | 3 %  |
| Dried milk            | 0.9% |
| Beet pulp             | 1.7% |
| Fish scrap            | 0.7% |
| Brewer's yeast        | 0.5% |
| Salt                  | 0.5% |
| Vitamins and minerals | 0.1% |



## Pet and Other Feeds

In this specific example, 100 parts by weight of those charge materials containing 9% water was placed within a Beale tube, where it was maintained for 2 minutes at 212°F. During this time, 10 parts of 70°F. water were added and steam was directly passed to the mixture. The temperature was maintained both by the steam passed directly to the mixture as well as by steam in the jacket. During the entire two minutes, the mixture was vigorously agitated. The moisture content of the material at the end of the 2 minutes was 30%.

The mixture was then placed in a Wenger extruder which extruded the so treated material at high pressure. The product, which came out of the extrusion die, was in the form of a rope having a density of 16 lb. per cubic foot. This material looked similar to angel food cake and had more-or-less oriented cells or air pockets. It was cut to desired length of about 3/4". The cut rope was then maintained for 10 minutes in the traveling bed drier through which hot air at 250°F. was passed. The dried particles were cooled by ambient air during a period of 3 minutes to give a product having a density of 18 to 20 lb./ft.<sup>3</sup>.

The so prepared particles at temperature of about 70°F. were placed in a rotating drum. Prime grade beef tallow at 125°F., in amount of about 4 lb. per 100 lb. of particles, was sprayed onto the particles as the drum rotated. Spraying was effected with a pump operating at 100 psig through a fine nozzle. Simultaneously and separately, a meat flavor stock (which had been prepared by heating beef in water for 2 to 3 hours) was sprayed onto the particles in amount of about 0.6 lb. per 100 lb. of particles.

When these had been added to the particles, a dry gravy-forming mixture which had been prepared by mixing about 1 lb. of precooked potato flour and 2 lb. of guar gum, was blown onto the particles. When the mixture was homogenous, mixing was stopped. One part by weight of this product was added to two parts by weight of warm (110°F.) water. The mixture was stirred for about 30 seconds to distribute the water throughout the mass. Gravy formed almost instantly. The mixture was not mushy or soggy; each of the individual particles of food retained its discreteness. The gravy-containing product was found to be very highly palatable when given to dogs.

### Semi-Moist Marbelized Meaty Food

D.P. Bone (U.S. Patent 3,380,832; April 30, 1968; assigned to The Quaker Oats Company) has a process which provides a meaty pet food, (i.e., one containing substantial amounts of meat or meat by-products) of the semi-moist type containing 20 to 50% moisture and having the appearance of marbled meat.

Example: This illustrates the manufacture of a beef-like marbled meaty pet food which does not melt at 135°F. The ingredients making up the base, and making up the marbling, were thoroughly mixed in separate blenders, in the proportion indicated in the table shown on the following page.

The material referred to as Base in the table results in a first sheet having the appearance of lean meat while the material referred to as Marbling in the table results in a second sheet having the appearance of fat. The base ingredients and the marbling ingredients were mixed separately and the resulting mixtures were charged to separate extruders at such a rate that the weight ratio of base extrudate to marbling extrudate was approximately 3:1.

# Pet and Other Feeds

|                                      |         | Percent  |               |
|--------------------------------------|---------|----------|---------------|
|                                      | Base    | Marbling | Total Formula |
| Beef Tripe                           | 29.0000 | 42.50    | 32.3750       |
| Sugar, Ready Mix Grade               | 26.5778 | 26.50    | 26.5584       |
| Sodium Caseinate                     | 15.0000 | 15.00    | 15.0000       |
| Beef Trimmings, 40% Lean             | 13.5000 | --       | 10.1250       |
| Corn Starch                          | 7.5000  | 7.50     | 7.5000        |
| Propylene Glycol                     | 4.5000  | 4.50     | 4.5000        |
| Dicalcium Phosphate, Dihydrate       | 2.5000  | 2.50     | 2.5000        |
| Salt, Iodized                        | 1.2000  | 1.20     | 1.2000        |
| Potassium Sorbate                    | 0.1000  | 0.10     | 0.1000        |
| Vitamin E Supplement (20,000 IU/lb.) | 0.0670  | --       | 0.0503        |
| Titanium Dioxide                     | --      | 0.20     | 0.0500        |
| Riboflavin Supplement (4 g./lb.)     | 0.0270  | --       | 0.0202        |
| Vitamin A Supplement (30,000 IU/lb.) | 0.0178  | --       | 0.0134        |
| Irradiated Dried Yeast               | 0.0040  | --       | 0.0030        |
| F D & C Red No. 2                    | 0.0027  | --       | 0.0020        |
| F D & C Yellow No. 6                 | 0.0027  | --       | 0.0020        |
| Thiamine Mononitrate                 | 0.0010  | --       | 0.0007        |

The conditions in the two extruders were substantially identical. The extruders were steam jacketed, and the combination of friction and externally applied steam heat resulted in the achievement of peak temperatures in the extruders in the range of approximately 260° to 275°F. The pressures utilized were adequate to prevent substantial expansion of the plastic mass within the extruder. Prior to expulsion from the extruder through the sheeting die, the compressed plastic mass was forced through a cooling section consisting of a pipe-like projection from the end of the extruder with the result that the temperature of the plastic mass at the die was substantially below the peak temperature range (260° to 275°F. referred to above).

This cooling step is desirable in that it minimized popping and spattering of the extrudate. The sheeting die utilized in the extrusion of the base provided an extrudate which was approximately 3 inches wide, and generally between 1/16 and 1/2 inch thick. The die utilized in the extrusion of the marbling sheet provided an extrudate sheet which was approximately 1 1/2 inch wide and likewise between about 1/16 and 1/2 inch in thickness. Upon leaving the extruder, the extrudate develops large bubbles, e.g., in the order of from 1/2 to 1 1/4 inch in diameter which pop leaving an irregular surface on the unexpanded extrudate.

The first sheet resulting from the extrusion of the base material was laid down on a moving conveyor belt and the second sheet resulting from the extrusion of the marbling ingredients was laid down on top of the first sheet on the conveyor belt. The second sheet was laid down, however, in an irregular and nonuniform pattern by rolling, skipping and balling portions of the second sheet prior to and/or upon its contact with the first sheet. The purpose of this is to randomize the distribution of the marbling sheet on the lean meat-like first sheet in such a manner that only about a fourth of the surface of the first sheet was covered by the fat-like extrudate. The conveyor carried the superimposed sheets for sufficient length to permit the temperatures of the extrudates to drop to approximately 150°F.



before the sheets were rolled into a roll. The roll was formed in such a manner as to provide the shape of a long loaf approximately 3 feet in length. As this loaf was continuously revolved around its longitudinal axis, the superimposed extrudate sheets coming off the conveyor were wound in random back and forth fashion until a loaf about 8 inches in diameter was formed. Several loaves of equal size were prepared in this manner. The extrusion of the lean meat-like portion was then terminated. The extrusion of the marbling portion continued, however, and each of the 3 foot by 8 inch loaves previously produced were given an overlay of marbling portion between about 1/4 and 3/4 of an inch in thickness by replacing the loaf in the rolling device below the end of the conveyor and wrapping the fat-like extrudate around the continuously rotating loaf.

These loaves were permitted to cool to approximately room temperature, at which time they were cut into steaks approximately 3/4 of an inch in thickness. However, to facilitate production rates, the loaves may be cut immediately after forming, as desired. The steaks resembled marbled beef in that the red, lean meat portion was marbled throughout in a random fashion by the fat-like portion. Steaks produced in a substantially identical manner were further cut into cubes approximately 1/2" x 1/2" x 3/4" and fed to dogs in a feeding test in which the fresh beef stew meat cut into cubes of substantially the same dimensions was used as a control. It was found that the product of this process ranked equal to the fresh beef stew meat with respect to acceptability by the animals.

## High Acid Content Palatable Dog Food

Pet foods in the process developed by F.J. Hallinan, E.J. Czarnetzky and A.I. Coombes (U.S. Patent 3,115,409; December 24, 1963; assigned to Wilson & Co., Inc.) are prepared by adding to the solid ingredients sufficient edible acid to produce a strongly acidified proteinaceous mixture. The added quantity of acid may be sufficient to produce a pH in the final cooked product of between about 2.0 and 5.0 or it may be only sufficient to produce a desired initial pH of about 4.5 to 5.0 for initiation of cooking with the intention that the pH be adjusted during the cooking operation to assure a proper final acidity.

The precooked pet food resulting from the acid-cook alteration of proteinaceous meaty material is packaged while still hot for storage and sale. A pet food of conventional moisture content was made from the following ingredients:

| <u>Ingredients</u>   | <u>Percent by Weight</u> |
|--|--------------------------|
| Water  | 43.14                    |
| Ground, cooked pork skins  | 30.00                    |
| Steamed bone meal  | 1.00                     |
| Lactic acid  | 2.50                     |
| Cracked barley   | 13.50                    |
| Soy grits  | 5.50                     |
| Wheat germ   | 3.00                     |
| Food supplements and flavoring (vitamins, cod liver oil, iron oxide, onion powder, etc.) | 1.36                     |

All of the ingredients except the grain source items (cracked barley, soy grits and wheat germ) were placed in a steamed jacketed mixer which was run for 15 seconds. The mixer was then turned off, but the ingredients were heated until the contents came to a boil. The grain source items were then added and the mixture was agitated for periods of 15 seconds at 3 minute intervals (to prevent scorching) until the temperature reached 200°F.

The heated product was then tightly packed in a paper container coated with a polyvinylidene chloride film, care being taken that there were no air pockets in the container. The container was then sealed. There was a small empty head space between the product and the container cover. The temperature of the product at the time of sealing was 180°F.

The container was then inverted so that the heated product came in contact with all of the inner surfaces of the container and the container and its contents were then allowed to cool to room temperature. The resulting product had a pH of about 4.0 and samples of the product which were opened a year after packaging had the same pH. The pet food was nutritious and appetizing to animals.

### Pelleted Dog Food Using Raw Cereal Grains

E.F. Linskey (U.S. Patent 3,139,342; June 30, 1964; assigned to Corn Products Company) has developed a process to prepare dog food in the form of small pellets of generally spherical form of a diameter of from 1/16" to 3/4". The surface of the pellet is relatively smooth and rounded, and is substantially free of dust or dust producing projections.

The interior of the pellet is cellular. It is produced in that form by virtue of the sudden release of external pressure upon small masses of the compressed material in a moist and heated condition. The steam puffs the small mass and causes it to assume a convex, rotund form. It is relatively smooth with some surface irregularities on the outside and highly porous on the interior. It then has a bulk density of about 15 to 21 lb. per cubic foot. Upon the relatively smooth surface there may be deposited, with considerable accuracy as to amount added, various taste enhancing and/or nutrition supplementing materials as coatings. This can be seen with reference to Figure 13.1.

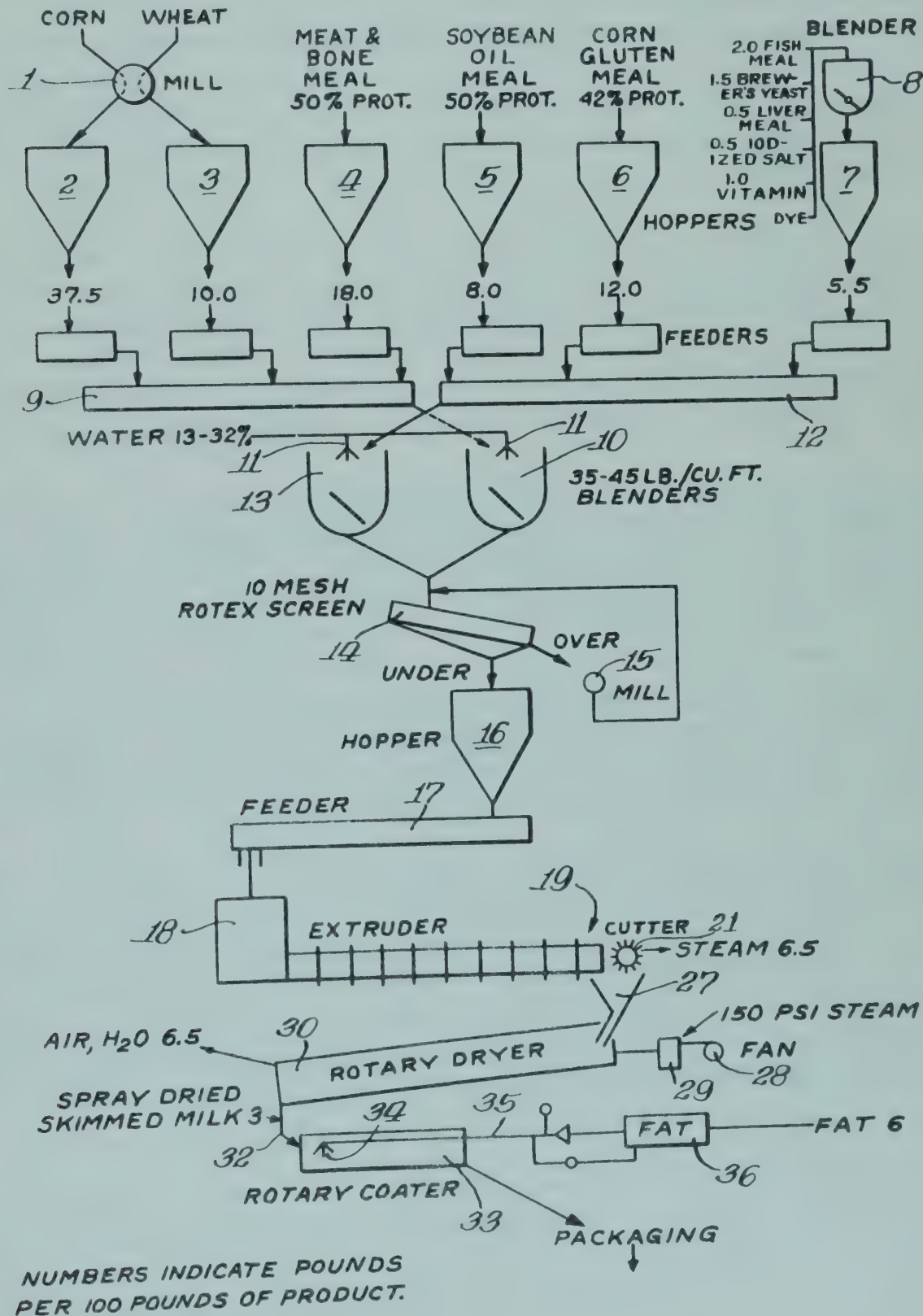
Corn and wheat are ground in the mill 1 and delivered to the hoppers 2 and 3, respectively, in the proportions indicated on the drawing. Middling starch from wheat shows the greatest puffing on expansion, and ground corn is next in order of that quality. This quality is desired in the mix delivered to the extruder. Meat and bone meal containing 50% protein are delivered to the hopper 4. Soya bean oil meal containing 50% protein is charged into the hopper 5. Corn gluten meal containing 42% protein is charged into the hopper 6, and the hopper 7 is charged with food supplement from the blender 8.

This supplement in the specific example includes 2.0 lb. of fish meal, 1.5 lb. of brewery yeast, 0.5 lb. of liver meal, 0.5 lb. of iodized salt, 1.0 lb. of dog vitamin and preferably sufficient dye added to the mixture to obtain from the extruder a suitably colored product. This may be a reddish brown color resembling the color of raw meat. These ingredients are blended in the blender 8 and then are delivered to the hopper 7. The blending of the various ingredients is effected as follows. Ingredients from hoppers 2 to 7 are metered into conveyors 9 and 12 in the proportions indicated on Figure 13.1. The joint discharge of material from



conveyors 9 and 12 is delivered alternately to hoppers 10 and 13. The discharge is being delivered into blender 13 as indicated by the full line arrow. While the ingredients are being mixed in the blender 13, or 10 as the case may be, water is added via spray nozzle 11 to raise the moisture to the desired level for extrusion.

FIGURE 13.1: PELLETED DOG FOOD USING RAW CEREAL GRAINS



When the mixing and moistening cycle of the contents of blender 13 is completed, the contents of that blender are discharged to the Rotex Screen 14. While blender 13 is discharging, ingredients from conveyors 9 and 12 are fed to blender 10 as indicated by the dotted line arrow to start a new cycle. The mixture delivered by the blenders 10, 13 will have an average bulk density of from 35 to 45 lb. per cubic foot. The mixture delivered by the blenders 10, 13 will have a moisture content of about 13 to 32% by weight.

The moistening of the material in the blenders 10, 13 to the degree indicated on Figure 13.1 and preferably to between 20 and 23% permits plasticization within the extruder 19 and provides the necessary moisture to be flashed into steam to produce the puffing or expanding operation. By way of example, material of a water content of approximately 20 to 23% when it enters the extruder 19 will lose about 6.5 lb. of water per 100 lb. of product by flashing off when it is extruded.

The material rejected by the screen 14 is sent through mill 15, and returned to the top of the screen. Material passing through the screen 14 enters the hopper 16. From the hopper 16, the material passes by way of the automatic feeder 17 to the intake 18 of the extruder 19. It comprises three main parts, the drum or barrel portion with orifice and inlet. The second part is the longitudinally extending screw which is contained in the barrel, and which is connected to a rotatable shaft mounted in bearings in a bearing frame.

The shaft is adapted to be driven by a suitable driving gear, not shown. The pitch of the screw is graduated so as to decrease the displacement of the material per turn of the screw as the material approaches the orifice. The orifice is served on the inside by the screw which forces material through the orifice to the outside. The third main part involved in forming the pellets is the multiblade cutter or knife 21 which is continuously driven during operation of the extruder by means not shown. The exit end of the orifice is served by the rotating multiblade knife 21 which cuts the issuing stream into short lengths of approximately the diameter of the orifice, so that when each separated piece is expanded fully by flashing of the contained moisture into steam, the small mass thus severed tends to assume a spherical shape and to increase in total volume by the expansion of the steam bubbles inside of the same.

The expanded pellets from the extruder 19 are exposed to the air to permit of free expansion and the escape of steam. They are then passed through a funnel 27 into a rotary drum or other suitable form of dryer. The pellets lose about 6.5 lb. of steam per 100 lb. of the product at the outlet end of the extruder 19. In passing slowly through the rotary dryer 30, which is an inclined rotating drum with a blast of air delivered therethrough from the fan 28 through a heater 29, the pellets lose a further 6.5 lb. of moisture per 100 lb. of product. The air from the fan 28 passes through the heater 29 which is heated by steam at about 150 lb. per square inch.

From the rotary dryer 30, the pellets with an additive of spray dried skimmed milk in the proportion of about 3 lb. per 100 lb. of the product are conveyed by the conveyor line 32 to the rotary coater 33. Other forms of coater may be employed. After the extrusion and drying operation, there may be added as a coating or a part thereof such materials as Vitamin A supplement which might be injured or destroyed by being subjected to the extrusion operation. Other coatings or flavor, color, etc., may also be added. Such coating operation may precede coating with fat or may follow the same. The firm, relatively smooth surface of the



pellets lends itself well to such coating treatment. The rotary coater 33 is a rotating drum at the inlet end of which there is a spray head 34 supplied with fat in liquid form through the pipe 35 from the supply tank 36. In this operation, about 6 lb. of fat per 100 lb. of product are added to the pellets. This is applied substantially as a coating. From the rotary coater 33, in which the fat is supplied at a temperature slightly higher than the melting point, the pellets are delivered to a packaging machine (not shown). Here the pellets are boxed in cardboard boxes or bagged in suitable bags ready for the shelf of the retailer. Analysis of the formula as shown in the flow diagram on a typical batch is as follows, as taken at two moisture levels: (Moisture is lost at the outlet end of the extruder 19).

|              |                  |                  |
|--------------|------------------|------------------|
| Moisture     | 8.3%             | 10 %             |
| Protein      | 27.1%            | 26.6%            |
| Crude fat    | 7.7%             | 7.6%             |
| Crude fiber  | 2.6%             | 2.5%             |
| Ash          | 7.6%             | 7.5%             |
| Starch       | 29.9%            | 29.3%            |
| NFE          | 46.7%            | 46.7%            |
| Bulk Density | 19.4 lb./cu. ft. | 19.8 lb./cu. ft. |

### Intermediate Moisture Type Food

A process for preparing an intermediate moisture type of dog and cat food, which is palatable, nutritious and can be stored and marketed under nonrefrigerated condition without the need for commercial sterilization in hermetically sealed packages has been developed by H.M. Burgess and R.W. Mellentin (U.S. Patent 3,202,514; August 24, 1965; assigned to General Foods Corporation).

Example: A specific example of the process is as follows: A mixture comprising 18.4 lb. of scalded beef tripe, 6.1 lb. of rough tongue gullets, and 6.1 lb. of beef cheek trimmings was placed in a double jacketed sigma mixer and heated to 212°F. with indirect steam over a period of 10 minutes. To the liquefied meat was added 2.0 lb. of propylene glycol, 0.3 lb. of potassium sorbate, 0.2 lb. of garlic oil, 2 lb. of tallow, 0.6 lb. of salt, 0.25 lb. of di-calcium phosphate, 0.6 lb. of vitamin premix, 0.001 lb. of cobalt sulfate and 0.005 lb. of red dye No. 2. Mixing continued during the addition of these ingredients and the temperature of the mix was maintained at about 200° to 212°F.

The liquid temperature was then reduced to about 160°F. after which untoasted soy flakes (3.15 lb.) were mixed in, and the resulting thick mixture was stirred for 5 to 10 minutes. Then, 25.9 lb. of Frodex (a commercial mixture containing 42% dextrose) and 5.1 lb. of dried skim milk were added to the mixture, and subsequently 3.5 lb. of flaked soybean hulls were added.

The mixture was thoroughly blended and then extruded through a low temperature, low pressure Enterprise extruder having a round 0.1875 inch diameter nozzle. The extruded cylinder was cut into lengths of about 0.5 inch and the lengths were then formed by low pressure into three-ounce patties having a diameter of 3.5 inches and a thickness of 0.75 inch. The so-prepared patties had a moisture content of about 25%, a pH of 6.8, a protein content of about 22%, and a fully balanced measure of other desired nutritional ingredients. It was

highly palatable and had an extended storage life even in the absence of refrigeration.

### Fat Coated Feed Annuli

M.A. Williams (U.S. Patent 3,284,211; November 8, 1966; assigned to Central Soya Company, Inc.) has developed a process to provide an animal food in which at least the surface is uniformly impregnated with fat which limits the development of crumbling and fines during handling, and packaging, and is more attractive to pets because all of the particles are uniformly impregnated.

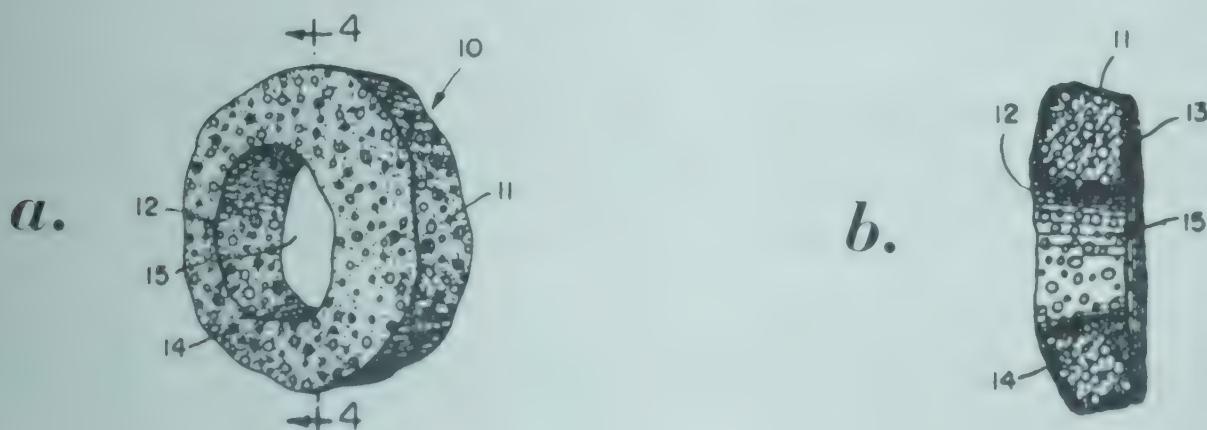
As seen in Figures 13.2a and 13.2b, the particle is generally in the shape of a cylindrical annulus 10, and is seen to include a generally cylindrical outer wall 11 and a generally cylindrical inner wall 12 bounded by top and bottom walls 13 and 14. One of the end walls as at 14 is generally convex, and the inner cylindrical wall 12 defines a hollow axial opening 15. Further, this defines a cross-sectional configuration that is generally rectangular.

The annulus 10 may be produced through an extrusion die where the die openings are of the order of 13/16" in diameter with the axial pin diameter for each opening being 7/16". This results in an annulus having an outer diameter following expansion from the die of the order of one inch and the bore diameter 15 being slightly less than the diameter of the pin resulting in the axial opening 15. Optimally, the thickness of the annulus is about one-half the radial width of the annular portion.

Example: An animal food mash including about 20 to 32% crude protein, 2 to 8% crude fiber, 5 to 15% ash, and 50 to 70% grain (the grain providing some of the protein), is heated for about 3 minutes, using 10 psi steam to develop a temperature in the cooker of the order of 230°F. The moisture content during this operation is increased to about 40% through the addition of water and the condensed steam.

Thereafter the cooked food passes through the extruder which also is equipped with the usual feed screw terminating in a die, the food expanding upon leaving the die and flash cooling to a moisture content of the order of 28 to 30%.

FIGURE 13.2: FAT COATED FEED ANNULI



Source: M.A. Williams; U.S. Patent 3,284,211; November 8, 1966



An exhaust fan adjacent the extrusion die is used to carry away the vaporized moisture and the heat generated by the extrusion, the die reaching a temperature of 330°F. and providing the latent heat of vaporization for the evaporated moisture. The expansion of the annular particle occurs primarily on the leading face 14 developing the convexity apparent in Figure 13.2b and it will be appreciated that the end faces 13 and 14 are relatively more porous than the inner and outer generally cylindrical surfaces 11 and 12, the latter having been compacted slightly more by the frictional engagement with the die.

The extensive surface provided by the annulus 10 develops a high rate of flash evaporation of the moisture which reduces the sticky or adhesive character of the annuli and the plasticity so that they can be intermixed in the chute without adhering together or deforming. On the other hand, sufficient moisture remains in each annulus as to give it an essentially resilient character whereby it can be handled without shattering, crumbling, etc.

The annuli are then transported by the bucket conveyor to the dryer and kept about 20 minutes, with the entering air temperature about 250° and 210°F. for the exiting air. This reduces the moisture content in the annuli to 11 to 12%, and generally in the range of 10 to 15%. Thereafter, in passing through the chute, the annuli which are partially dried, enter the tumbler which is essentially a tilted cylindrical rotatable mechanism equipped with interior sprays where the annuli have a temperature of the order of 130°F. and where the sprays add about 3% prime tallow which is liquefied by heating to a temperature of about 130°F. The prime tallow or fat attractant is beneficially fortified with vitamin A. The fat has a congealing temperature of the order of 96°F. and that making a temperature suitably above this level develops advantageous coverage of the annuli.

The tumbler is of relatively large diameter, of the order of 3 to 5 feet, so that the annular particles for the most part rest on what can be considered flat surfaces, with the particles disposed end downwardly, i.e., lying on the end surfaces 13 and 14. These are the more porous surfaces and are usually alternately exposed by virtue of the tumbling so that even though each end surface is exposed only approximately one-half the time, there is ample opportunity for the prime tallow to penetrate by virtue of the greater porosity of these end surfaces.

Thereafter, the annular particles are conducted through the chute to a single pass cooler which again is equipped with a conveyor belt. The residence time in the cooler is of the order of 2 minutes at room temperature, where the moisture content of the particles is reduced to the range of 8 1/2 to 9 1/2%. This results in a product discharged through the bagging machine which meets the following specifications:

- Not less than 24% protein
- Not less than 7% fat
- Not more than 4.5% crude fiber
- Not more than 10% ash
- Not more than 12% moisture

Additionally, the particles have a bulk density of 18 to 21 lb. per cubic foot, and experience has shown that less than 3% of the weight of the particles passes through a 1/2" mesh screen.

# Acetamide Flavor Enhancer

H.H. Young (U.S. Patent 3,203,806; August 31, 1965; assigned to Swift & Company) has a process which provides pet foods which possess a greater pet acceptance. The flavor enhancing additive contains acetamides as an essential ingredient. Crude or impure acetamide containing a small amount of acetic acid and a mixture of mono-, di- and triacetamide is particularly effective.

Example: Wet, dry and semidry pet foods contain the following ingredients in the amounts noted:

| Ingredient  | Wet     | Dry     | Semidry |
|---|---------|---------|---------|
| Meat, meat by-products and other animal protein, percent..... | 20-35   | 10-40   | 30-50   |
| Fat, percent.....   | 1.0-6.0 | 4.5-9.0 | 10-25   |
| Cereals (total breakdown), percent.....                       | 10-20   | 70-90   | 30-60   |
| Soybean grits or meal, percent.....                           | 8-12    | 25-40   | 10-15   |
| Wheat and/or barley, percent.....                             | 3-8     |         | 10-15   |
| Corn and/or milo, percent.....                                | 2-5     | 30-60   | 10-15   |
| Others, percent.....  | 0.5-2.0 | 2-5     | 2-8     |
| Minerals, in percent:   |         |         |         |
| Calcium (minimum).....  | 0.4     | 1.0     | 0.8     |
| Phosphorus (minimum).....                                     | 0.3     | 0.8     | 0.6     |
| Potassium (minimum).....                                      | 0.3     | 0.8     | 0.6     |
| NaCl (minimum).....   | 0.5*    | 1.4*    | 1.0*    |
| Minerals, in percent:   |         |         |         |
| Iron.....   | 8.0     | 22      | 16      |
| Copper.....   | 1.0     | 2.5     | 2.0     |
| Magnesium.....  | 70.0    | 200     | 140     |
| Manganese.....  | 0.7     | 2.0     | 1.4     |
| Iodine.....   | 0.2     | 0.5     | 0.4     |
| Vitamins:   |         |         |         |
| Vit. A units/lb.....  | 600     | 600     | 600     |
| Vit. D units/lb.....  | 80*     | 120*    | 100*    |
| Vit. B12.....   | 0.004   | 0.01    | 0.008   |
| Thiamine.....   | 0.1     | 0.3     | 0.2     |
| Riboflavin.....   | 0.3     | 0.8     | 0.6     |
| Pyridoxine.....   | 0.15    | 0.4     | 0.3     |
| Pantothenic acid.....   | 0.4     | 0.9     | 0.8     |
| Niacin.....   | 1.5     | 4.1     | 3.0     |
| Choline.....  | 200     | 500     | 400     |

\*In mg./lb. of feed.

In the case of wet foods, the additive in the amount of 0.2 to 0.8% based on the solids present in the pet food is incorporated into the ingredients during the formulation thereof. With dry or semidry pet foods, the additive is conveniently sprayed on the dry food from an aqueous solution just prior to packaging and at lower levels from 0.1 to 0.5%. The wet food was employed in dog acceptance tests using sixteen dogs for three consecutive days. This laboratory test showed the following results:

| Sample                            | Preference |         |
|-----------------------------------|------------|---------|
|                                   | No.        | Percent |
| Control                           | 13         | 27      |
| Control containing 0.2% acetamide | 20         | 41.7    |

Fifteen dogs, or 31.3%, showed no preference. Similar results are obtained when the additive is incorporated in cat food containing a large amount of fish.



## COMPOSITIONS FOR BIRDS

### Diet Supplement for Caged Pet Birds

A composition for the treatment of caged pet birds is described by E.J. Ross and C.H. Elbreder (U.S. Patent 2,946,685; July 26, 1960).

Example 1 — Chemical Preparation: A formula is listed below in which a binder of sucrose to provide palatability is employed.

| Component                     | Amount                                   | Percent by Weight |
|-------------------------------|--|-------------------|
| Streptomycin.....             | 10 mg.....                               | 5.15              |
| Penicillin.....               | 3,000 units (1.9 mg.) <sup>1</sup> ..... | 0.98              |
| Vitamin B <sub>12</sub> ..... | 0.1 mcg.....                             | 0.0000515         |
| Sucrose.....                  | 167 mg.....                              | 85.86             |
| Magnesium Silicate.....       | 15.5 mg.....                             | 8.0               |

<sup>1</sup> 627 grams equivalent to 1 billion units.

Such tablets when made in the weight of 3 grains (194 mg.) are added in amounts of about 1 tablet to each ounce of bird's daily drinking water. As an example for the application to parakeets, which ingest approximately one ml. of water per day, such a dosage provides approximately 0.003 mcg. of vitamin B<sub>12</sub>, 100 units of penicillin and 330 mcg. of streptomycin sulfate. The composition has been found valuable to increase general health and provide weight gain as shown below.

| Subject                | Treatment  | Results  |
|------------------------|--|--|
| Cage #1, 1 Canary..... | 1,500 Units Penicillin G. Potassium plus Vitamin B <sub>12</sub> 0.1 mcg.  | 5 Days 17% gain.<br>15 Days 20% gain.<br>30 Days 23% gain. |
| Cage #2, 1 Canary..... | 1,500 Units Penicillin G. Potassium plus 15 mgm. Streptomycin Sulfate base plus Vitamin B <sub>12</sub> 0.1 mcg. | 5 Days 19% gain.<br>15 Days 20% gain.<br>30 Days 25% gain. |
| Cage #3, 1 Canary..... | Control—No antibiotic used.  | 5 Days 9% gain.<br>15 Days 12% gain.<br>30 Days 15% gain.  |

It is useful in curing common respiratory diseases, arthritis and bacterial enteritis. Tests were run with sick birds.

Example 2: When 3,000 units of penicillin G potassium were employed with 0.1 mcg. of vitamin B<sub>12</sub> per ounce of drinking water out of a total of 6 birds treated 3 were lost and this resulted in a 50% curing of the birds. Where 10 mgm. of streptomycin sulfate base were employed, out of a total of 6 birds treated, only 1 bird was lost and 84% were saved. Where a total of 6 birds were treated employing the chemical preparation of this process using 3,000 units penicillin G potassium plus 10 mgm. streptomycin sulfate base plus 0.1 mcg. of vitamin B<sub>12</sub>, no birds were lost and all 6 birds were saved for a 100% rating. Where a control was employed signifying no treatment whatsoever, out of the 9 birds tested, 8 were lost and thus only 12% were saved.

Introducing Physiologically Active Material into Bird Seed

A process for introducing physiologically active materials, into bird seed, in which a substantial proportion of the materials has penetrated to the kernel of the seed has been developed by H. Rabinovitch (U.S. Patent 3,136,640; June 9, 1964; assigned to Laboratories for Applied Biology Limited, England).

Example: The husk of bird seed is first split by either of the following methods:

- Method A. 12 g. of canary seed was added to 25 ml. of boiling water and boiling was continued for 10 minutes. The seed was separated from the water and dried at a temperature not exceeding 50°C.
- Method B. 12 g. of canary seed was supported on a perforated disc placed in a vertical cylindrical vessel and steam was passed therethrough for from 10 to 15 minutes. The seed was then removed.

Seed treated by Method A above was added to 96% ethyl alcohol and a concentrated solution of vitamin B<sub>1</sub> in 96% alcohol was added, the amount of vitamin being such that there was approximately 1 mg. of vitamin for each 100 g. of seed. The seed was allowed to stand in the alcoholic solution for 24 hours and the alcohol was then decanted off and the seed allowed to stand in the air so that the alcohol evaporated from it.

In an alternative process removal of the alcohol from the seed was effected with the aid of vacuum. Instead of vitamin B<sub>1</sub>, vitamin B<sub>12</sub> was employed or a mixture of both vitamins was used. Other tests have been carried out in which vitamin A, the vitamin B complex, vitamin D and vitamin E have been used. In further examples various materials including specific antibiotics and other drugs have been introduced into seed, which seed has been successfully used in the therapeutic treatment of birds in captivity.

Molded Cuttlebone

Cuttlebone is used as a bird food and serves as a source of calcium and protein as well as a means by which the bird sharpens its beak. A.V. Hinton and M. Gerzanich (U.S. Patent 3,256,093; June 14, 1966) have a process for preparing a rigid molden bird food product comprising cuttlebone of powdery fineness embedded in a proteinaceous matrix.

The bleached powdered cuttlebone may be prepared by soaking the natural bone in a solution of approximately 2% hydrogen peroxide for a period of about one to two weeks, i.e., long enough to remove much of the natural oils, proteinaceous materials, fats and salts from the bone. After bleaching, the bone is thoroughly rinsed to remove the separated unwanted materials as well as any residue of hydrogen peroxide.

The washed, bleached cuttlebone is air dried and then ground into discrete particles to a size that will not settle out of a solution of proteinaceous material thickened by beating as will hereinafter be described, and additionally the particles must not be discolored in the act of grinding (40 to 50 mesh silk screen).



Example: A good molded cuttlebone made in accordance with the process is as follows:

|                              | <u>Parts by Weight</u> |
|------------------------------|------------------------|
| Bleached powdered cuttlebone | 192                    |
| Gelatin                      | 24                     |
| Egg albumin                  | 1                      |
| Cornstarch (gelling type)    | 16                     |
| Salt                         | 2                      |
| Water                        | 192                    |

Mix the cornstarch, egg albumin, and salt with 32 parts of cold water to form a paste. Pre-soften the gelatin by mixing it with 64 parts of water. Any water temperature from cold to boiling will suffice. Dissolve the presoftened gelatin mixture in 96 parts of boiling water and add to it the cornstarch, egg albumin, and salt mixture. Cool to a temperature between 100° and 105°F. and beat (usually about 1 minute) until mixture has a consistency which is self-supporting. Add the powdered cuttlebone to the mixture gradually and continue beating for about 1 minute or until a homogenous mixture is secured. Fill prechilled molds with homogenous mixture and refrigerate at a temperature within the range 2° to 10°F., preferably 6° for a period of from 10 to 30 minutes. 15 minutes of refrigeration is usually long enough to set the product sufficiently to permit its removal from the mold.

Upon its removal from the mold and air drying, a hard bonelike structure results comprising cuttlebone particles in a proteinaceous matrix, it being understood that the gelatin and the albumin used in the manufacture of the product are essentially protein. Air drying usually takes about 12 to 18 hours. Initial drying is at room temperature until a hard coating is formed. Thereafter drying preferably is carried out at a temperature of about 90° to 100°F. Maintenance of this second stage drying temperature constant eliminates some curling which otherwise seems to occur if the drying temperature during this stage is allowed to vary. However, except for such curling, the product is otherwise the same.

### Pecking Stone for Birds

R. St. Hilaire (U.S. Patent 3,293,039; December 20, 1966; assigned 50% to Vasilije Starcev) has developed a process to provide an improved grit cake for use by parakeets and like domesticated birds for the care and abrading of the bill and for supplying the necessary gravel, roughage and certain nutrients which captive birds frequently lack in their diet.

Example: In accordance with the process, the composition from which the cake or stone is molded to provide a relatively hard homogeneous mass of the preferred shape and size is as follows:

|                              |           |
|------------------------------|-----------|
| Crushed divided oyster shell | 2 1/2 oz. |
| Fine gravel (1/8" max. size) | 2 1/2 oz. |
| Finely divided charcoal      | 10 g.     |
| Powdered pumice              | 1 oz.     |
| Glastone                     | 2 oz.     |
| Honey (liquid)               | 1 1/2 oz. |

(continued)

(continued)

|                        |       |
|------------------------|-------|
| Cod liver oil (liquid) | 6 g.  |
| Table salt (NaCl)      | 4 g.  |
| Water                  | 2 oz. |

Glastone is a well known commercially available casting or molding composition employed in the dental art to produce a dental stone cast having improved compressive strength. It is employed in the process to bind the other ingredients together at the time of molding of the product.

The above ingredients in no special order are placed together in a receptacle and thoroughly mixed into a putty-like mass and are then placed in a conventional mold of desired shape and allowed to harden at room temperature 70°F. for approximately 2 1/2 hours. At the end of this time, a cake or stone is produced which is very hard and homogeneous and gray in color with individual particles of shell and gravel exposed at the surfaces of the stone and throughout the same.

By supplementing a conventional seed diet with the pecking stone, parakeets and like birds are maintained in excellent health and will devote their energies to pecking the stone instead of paint or other ornamental objects in the home.

## FISH BAITS

### Water-Soluble Fish Bait

G.A. Walldov (U.S. Patent 2,874,048; February 17, 1959) has a process for manufacturing fish bait which are slowly soluble in water to simulate bleeding of the bait, thus to make the same highly attractive to game fish. This is described with reference to the figure in which Figure 13.3a is a perspective view of a fish bait. Figure 13.3b is a perspective view of the bait on the reduced scale, as it appears when in use. Figure 13.3c is a transverse sectional view substantially on line 3—3 of Figure 13.3a. Figure 13.3d is a longitudinal sectional view through the bait substantially on line 4—4 of Figure 13.3a; and Figures 13.3e to 13.3h inclusive are perspective views showing the manufacture of the bait during succeeding steps of the process.

Referring to the drawings in detail, the bait has been designated at 10. In the illustrated example, the bait is shaped to simulate a worm, but could be a minnow, a frog or other bait animal. The illustrated fish bait comprises a body or carrier which includes superposed, identically shaped laminations 12, 14, separated by and adhering to a fabric binder strip 16 that constitutes an intermediate lamination. The binder is of an outer configuration matching that of the laminations 12, 14, which are formed of the water-soluble material. Dispersed through the laminations 12, 14 is a dye 17, the particles of which have been exaggerated in the drawing for the purpose of clearly showing this feature.

When the bait is impaled upon a hook 20 connected to a fishing line 22, it has the adaptability of attracting game fish in the vicinity, by reason of both the coloring and shape thereof, and on dissolving produces a dyed effusion 23 in which are contained the particles 17.



In this connection, the provision of the fabric binder 16 adapts the bait to be impaled upon a hook, and to remain in position upon the hook, despite the fact that the substance of which the laminations 12, 14 are formed is soluble in water. Gelatin, when combined with glycerin and other ingredients according to the process described, will retain its shape throughout the interval between manufacture and use, will also retain its shape throughout the period that the lure is in the process of dissolving, will be sufficiently soft to permit its being readily impaled upon a hook, will dissolve at a comparatively slow rate, and will be characterized by its cohesiveness and lack of tackiness.

Example: The process whereby the lure is made has been illustrated in Figures 13.3e to 13.3h, and in carrying out the process, ingredients are used as described:

|                            | <u>Parts by Weight</u> |
|----------------------------|------------------------|
| Gelatin                    | 64                     |
| Glycerin                   | 8                      |
| Dye                        | 1                      |
| Flavoring (odor-producing) | 4                      |
| Water                      | 128                    |

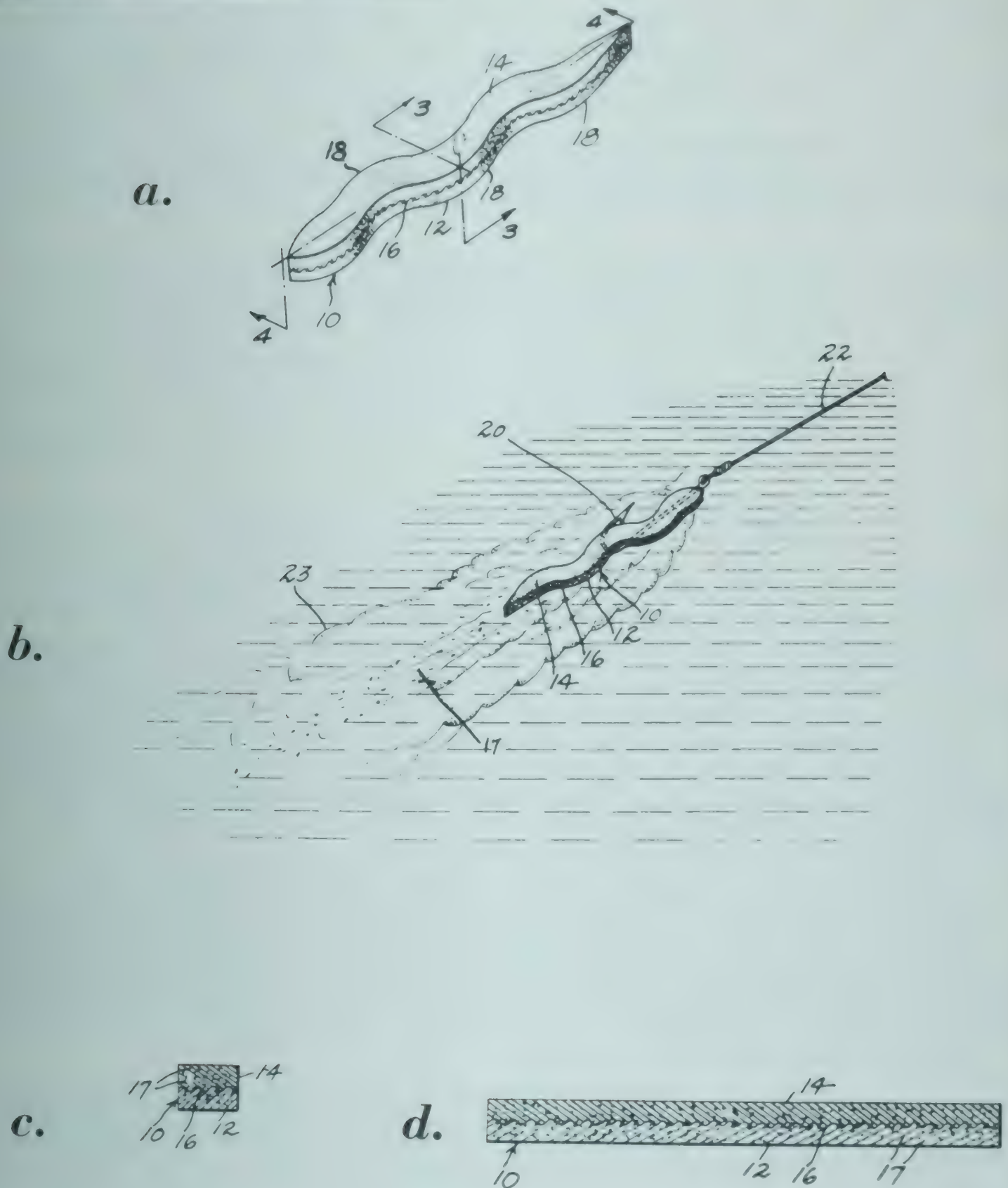
The gelatin used will be ordinary gelatin, which is commercially produced in a powder. A relatively high quality of glycerin has been found to be the most satisfactory. Ordinary vegetable dye is employed to advantage in the process, and can be of any color. For example, a red dye may be used to cause a blood-simulating cloud or slick when dispersed through the water on dissolving of the gelatinous substance of which the laminations 12, 14 are formed. When a white or milky effusion is desired, it has been found that powdered milk is entirely satisfactory and in fact, in the dissolving of the fish lure, the powdered milk tends to come off in flakes, so that there are small solids mixed in the effusion or slick 23, which solids are attractive to fish.

As to flavoring, anise, in powdered form, has been found to be entirely satisfactory. Commercially produced essences sold to fishermen and having anise bases are also satisfactory. A batch is preferably mixed in a double boiler. As a first step, the gelatin is mixed with the water while the water is still cold, since this tends to dissolve the gelatin more quickly. Heat is applied, until the gelatin is fully dissolved. The water temperature at which full dissolving occurs preferably is in an optimum range of 90° to 110°F.

After the gelatin has been fully dissolved, the remaining ingredients are added. The water temperature is kept at a level that will prevent solidifying of the gelatin while the other ingredients are being fully dispersed therethrough. The solution is then poured into a tray 24 as shown in Figure 13.3e, to provide a bottom layer 26 of the carrier material. When the solution in the tray begins to solidify, a cheese cloth 28 (Figure 13.3f) is placed thereon, but it will be understood that the cheese cloth is not applied until such time as it will remain upon but will be fully embedded in the top surface of the layer 26.

Thereafter, a time interval occurs during which there is further solidification of the bottom layer, to an extent such that when the top layer is applied, it will not press the cheese cloth down into the bottom layer any further than it has already been embedded.

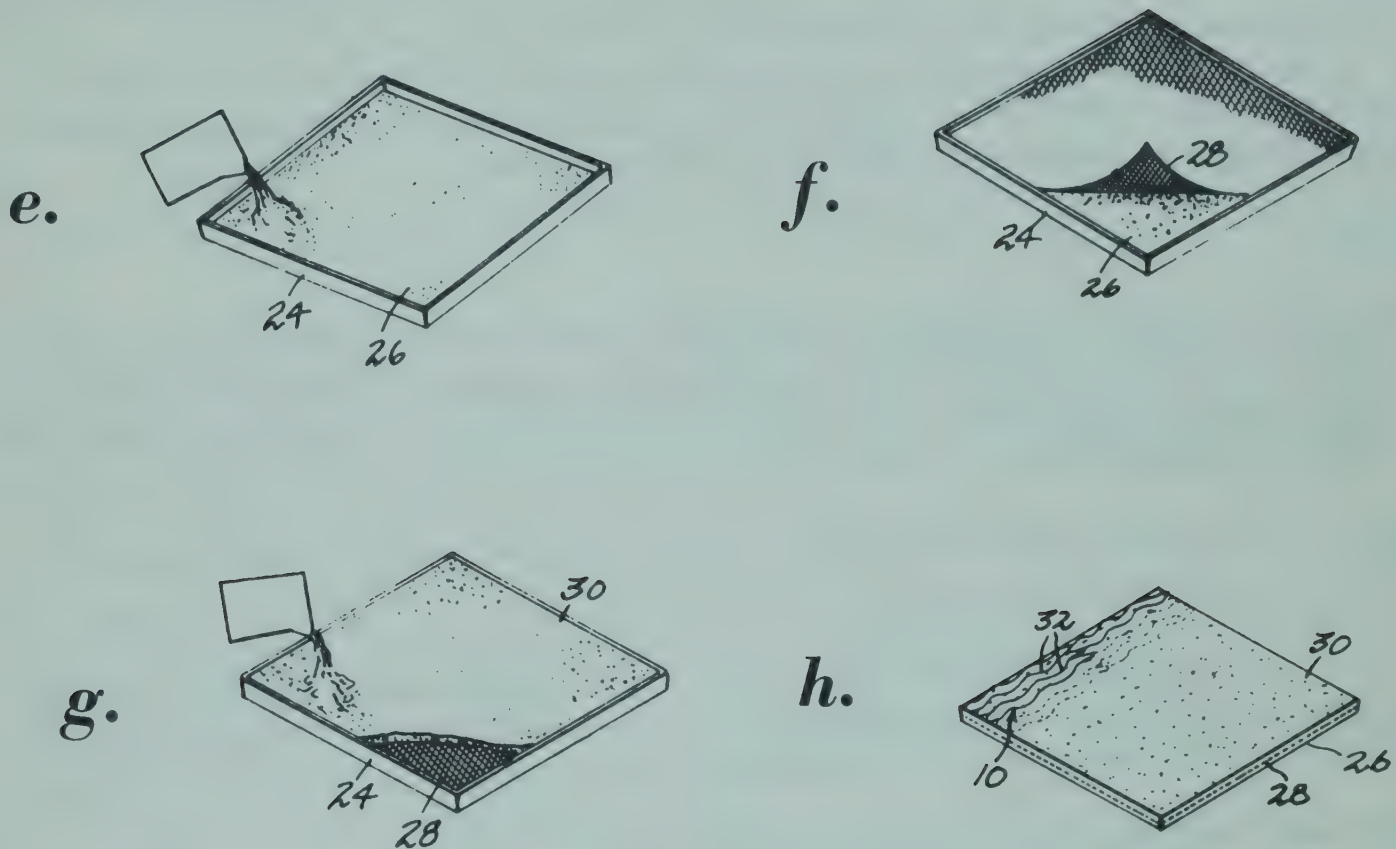
FIGURE 13.3: WATER-SOLUBLE FISH BAIT



(continued)



FIGURE 13.3 (continued)



Source: G.A. Walldov; U.S. Patent 2,874,048; February 17, 1959

Thereafter, a top layer 30 of the mixture, in a fully liquid state, is poured into the tray as shown in Figure 13.3g. Then, the top layer is permitted to solidify until both layers have become completely solidified. When the upper layer is solidifying, it will in turn bond to the fabric lamination so that in effect, both laminations are bonded together by the fabric lamination disposed between the same.

As a next step, the product is subjected to refrigeration at a temperature above freezing, as for example at approximately 42°F. to accelerate solidification and provide for easier handling of the material during further steps of the process. Thereafter, the product is subjected to dehydration. By means of a blower or fan, so arranged that the air currents generated thereby impinge directly upon the surface of the substance. In this connection, it is highly desirable that the impinging air be characterized by relatively low humidity, and it has been found that good results are obtained, in the absence of a dehumidification chamber, by directing the generated air currents over an exposed resistance element prior to their impingement upon the product.

Dehydration is continued until the product is capable of removal from the tray without adherence to the surface thereof, and has cohesion sufficient to permit easy handling and clean

## Pet and Other Feeds

cutting thereof during a die cutting operation that follows as the next step. The individual baits are now cut out, after removal of the flat, laminated, cake-like product from the tray, by a die cutting operation. This is shown in Figure 13.3h. The dies, not shown, would be of any desired shape, to produce the finished shape of the bait. The individual baits are further dehydrated, in the manner previously described, for easier handling and improved keeping qualities, to any extent preferred by the manufacturer.

### Dough Balls

S. Yakstis (U.S. Patent 3,322,544; May 30, 1967) has developed a method of preparing a fish bait which may be stored in a dry condition for long periods of time and then be readily adapted for use in fishing. The process is directed to preparation of a fish bait commonly known as dough balls.

Example: In accordance with the process the following quantities and ingredients were utilized:

|                                   |                                   |
|-----------------------------------|-----------------------------------|
| 2 tablespoons of corn meal        | 8 drops of vanilla extract        |
| 2 tablespoons of white flour      | 8 drops of food coloring (yellow) |
| 2 tablespoons of granulated sugar |                                   |

The corn meal, vanilla extract and food coloring are mixed together and then the sugar and white flour are added to this mixture and thoroughly mixed to form a homogeneous mixture of ingredients.

The ingredients are then packed into a cloth bag. The ingredients were then stored for several weeks. Following storage, the cloth bag was immersed in boiling water for 10 minutes. When the bag was removed from the boiling water, and the cloth bag cut away, the ingredients were formed into a paste-like material of relatively high viscosity. This paste-like material was formed into dough balls and was utilized by placing the dough balls over fish hooks to fish successfully for catfish and carp. The size of the dough ball will be determined by the size of the fish hook that it is to cover.

### Freeze Dried Unified Mass

A means of feeding the fish in an aquarium or other relatively limited environment, which would provide sufficient food for a single periodic feeding of the fish without, however, introducing excess food which would decay and tend to foul the water is described in the process of H.R. Axelrod (U.S. Patent 3,361,566; January 2, 1968; assigned to T.F.H. Publications, Inc.).

This is based on the concept of feeding fish or other aquatic animals by providing a unified mass of fish food, capable of being torn apart by feeding fish, adhered to a suitable surface positioned so that the adhered mass of food is accessible to the feeding fish. In this way fish are enabled to feed from the mass of fish food by tearing off and consuming small pieces until the mass of food is substantially completely consumed.

Example: A freeze dried unified mass of fish food which will adhere to a wetted surface



such as glass without added adhesive is prepared as follows: Living tubificid worms (*Tubifex* sp.), which are commonly known to aquarists as tubifex worms, are collected in their native habitat. As is well known, such worms are found in rivers throughout the world, existing in large colonies in mud immediately adjacent to areas rich in sewage. Such worms are an excellent source of protein, are avidly devoured by fish, and are of such small size that they may be eaten by all but very small young fish. The live tubifex worms are placed in clean running water for a period of about 72 hours at the end of which time the worms have emptied their digestive tracts.

Although the worms can be freeze dried immediately upon collection, they usually contain undesirably large amounts of foreign matter which is preferably removed. The living worms are then placed in suitable shallow containers such as pans about 3 feet square. The pans are then placed in a conventional low temperature freezer and quick frozen by subjecting them to a temperature of 0°F. for about 2 hours.

The pans of quick frozen worms are then placed in a conventional vacuum dryer and subjected to a temperature of about -25°C. and a vacuum of about 0.1 to 0.5 mm. of Hg pressure. While in the vacuum dryer the frozen worms are subjected to ultraviolet radiation to sterilize the worms and kill any harmful bacteria which the worms might be carrying in order to prevent growth of these harmful organisms in the aquarium or other environment to which the fish food is ultimately introduced.

The vacuum drying of the frozen worms is continued until a desiccated sheet of intertwined worms containing about 5% by weight or less moisture is obtained. The depth of live worms loaded into the pans is such as to produce a dried sheet about 1/8" to 1" or preferably about 1/4" to 1/2" in thickness. Thinner or thicker sheets may be produced if desired but sheets thinner than 1/4", especially below 1/8 or 3/16 inch in thickness tend to have poor mechanical strength and are, therefore, more difficult to handle and portion without waste.

Sheets more than 1/2" thick may be prepared but it is seldom necessary to do so since sheets of 1/4 to 1/2 inch in thickness generally provide a sufficiently large portion of worms for a single feeding for aquarium purposes, when cut into pieces of about 1/2 to 2 square inches in area. After cutting the desiccated sheet of worms into portions of desired sizes, these portions are placed in plastic containers and subjected to treatment with either nitrogen or carbon dioxide at about 50 psig at room temperature. In order to kill *Salmonella* which may infest the fish food it is desirable to subject the material to 80°C. heat for about 30 minutes. The resulting chips or pieces of desiccated tubifex worms are then placed in plastic bottles with screw caps for shipment and storage until used.

Packaging of this or a similar type, which limits access of moisture to the fish food, is desirable, since freeze dried fish food is normally hygroscopic. The chips produced in this way when immersed in water in an aquarium and pressed against the wet inner surface of one of its glass sides, will adhere to the glass sufficiently tightly to remain in place while the fish in the aquarium voraciously attack the chip, tugging at and removing morsels therefrom. The pressure required to cause adhesion of such chips of desiccated fish food is only light to moderate manual or digital pressure. The process can be followed using, alone or in combination, live brine shrimp, fresh ground beef muscle tissue, beef heart or liver or earthworms.

SILKWORM FEEDSEating-Inducing Compositions

The silkworm is monophagous in eating nothing but mulberry leaves. As a result of extensive studies Y. Hamamura, K. Hayashiya and K. Naito (U.S. Patent 3,275,446; September 27, 1966; assigned one-half to Yasuji Hamamura and one-half to Takeda Chemical Industries, Ltd., Japan) found factors in mulberry leaves responsible for this. They discovered that terpenes are the attracting factor; beta-sitosterol with or without flavonoids is the biting factor; and cellulose powder is the swallowing factor.

Example: A composition for inducing silkworms to eat comprises the following ingredients in approximately the following relative proportions by weight based on the total weight of the composition.

|                     | <u>Percent</u> |
|---------------------|----------------|
| Beta-sitosterol     | 0.1 to 1       |
| Cellulose powder    | 20 to 60       |
| Sugar               | 1 to 10        |
| Inorganic phosphate | 0.2 to 2       |
| Terpene             | 0.001 to 0.01  |
| Flavanoid           | 0.1 to 0.3     |

The terpene can be omitted when the silkworms are placed on the feed and hence when no attractant is required. For the practical feeding, addition of auxiliary factors may come into consideration.

Feed Intake Promoting Factor

In later work, Y. Hamamura, K. Hayashiya and K. Naito (U.S. Patent 3,328,170; June 27, 1967; assigned to Y. Hamamura, Japan) found that when feedstuff containing polyhydroxy-carboxylic acid, a feed intake promoting factor, is given to silkworms, the amount of feedstuff eaten by silkworms is effectively increased.

As polyhydroxy-carboxylic acid, there may be used chlorogenic acid, caffeic acid, gallic acid, gentisic acid, homogentisic acid, resorcylic acid, quinic acid, uronic acid, etc. Especially, employment of chlorogenic acid and/or caffeic acid gives a preferable result in general. Chlorogenic acid may be extracted from plants such as mulberry leaves, tobacco plant leaves, pear leaves, tea plant leaves, apple sarcocarp or chlorogenic acid produced by microorganisms, e.g. Piricularia oryzae Cava, Ceratostomella fimbriata Ellis may similarly be employed.

Example 1: Feedstuff for silkworms is prepared by mixing 1 g. of cellulose powder, 5 mg. of inositol, 10 mg. of inorganic phosphate, 0.3 mg. of chlorogenic acid and 5 mg. of beta-sitosterol with 3 cubic centimeters of water containing 2% of agar-agar and 3% of sugar.

Example 2: Feedstuff for silkworms consists of 5 g. of cellulose powder, 2.0 g. of defatted soybean powder, 1.5 g. of starch, 1.0 g. of sugar, 0.090 g. of Wesson's minerals, 0.04 g.



## Pet and Other Feeds

of vitamin C, 0.01 mg. of vitamin B<sub>1</sub>, 0.01 mg. of vitamin B<sub>2</sub>, 0.01 mg. of vitamin B<sub>6</sub>, 0.02 mg. of nicotinic acid, 0.02 mg. of calcium pantothenate, 0.002 mg. of folic acid, 0.002 mg. of biotin, 0.01 mg. of vitamin B<sub>12</sub>, 50 mg. of beta-sitosterol, 25 mg. of morin, 50 mg. of inositol, 50 mg. of K<sub>2</sub>HPO<sub>4</sub>, 250 mg. of SiO<sub>2</sub>, 10 mg. of chlorogenic acid and 15 cc of water.

### Propionic Acid as Growth Accelerator

K. Kato, S. Ide and K. Okada (U.S. Patent 3,295,983; January 3, 1967; assigned to Ajinomoto Co., Inc. and Katakura Industry Co., Ltd., Japan) have discovered that the growth rate of silkworm larvae fed on artificial feed is accelerated when the feed contains propionic acid and/or its salts, such as potassium propionate, sodium propionate, magnesium propionate, calcium propionate or zinc propionate.

Example: Composition of basic feed: mulberry leaf powder 48%, potato starch 15%, defatted soybean meal 10%, cellulose powder 20%, glucose 5%, ascorbic acid 2%, and necessary quantity of vitamin B group. 0% (comparison), 0.05, 0.1, 0.2, 0.4, 0.8, 1.6, 3.2% by weight of calcium propionate were respectively added to batches of the feed and each batch was kneaded with 1.5 times its weight of water, heated with steam for 10 minutes and cut into slices.

Newly hatched silkworm larvae were reared on the feed at a temperature of 25°C. and at 75% relative humidity for 15 days. Forty silkworms were tested for each experiment. The growth rate percentage and average weight of surviving larvae is shown in the table below.

| Amount of calcium propionate added (percent) | Percentage and average weight of surviving larvae |              |               |              |
|--|---|--------------|---------------|--------------|
|  | After 10 days                                     |              | After 15 days |              |
|  | Percent   | Av. wt., mg. | Percent       | Av. wt., mg. |
| 0  | 100   | 37.9         | 75            | 78.7         |
| 0.05   | 90  | 38.7         | 80            | 88.0         |
| 0.1  | 95  | 46.1         | 90            | 116.8        |
| 0.2  | 100   | 53.4         | 95            | 138.0        |
| 0.4  | 100   | 53.0         | 85            | 102.1        |
| 0.8  | 100   | 53.0         | 85            | 100.3        |
| 1.6  | 100   | 50.2         | 80            | 98.4         |
| 3.2  | 95  | 40.1         | 80            | 82.2         |

## STERILE ANIMAL FOOD

### Sterilizing Food in Sealed Container

Sterile food and other materials are particularly necessary in many fields of research, for example, for use in the study of experimentally produced diseases. Laboratory animals, such as hamsters, rats, mice, guinea pigs, chickens, dogs, pigs, monkeys etc. are used in this research, and it is vital that the animals be fed sterile food and maintained in a controlled environment.

J. J. Landy (U.S. Patent 3,215,539; November 2, 1965) provides a method where the materials are sterilized in a vapor-tight container of a form suitable for marketing. The

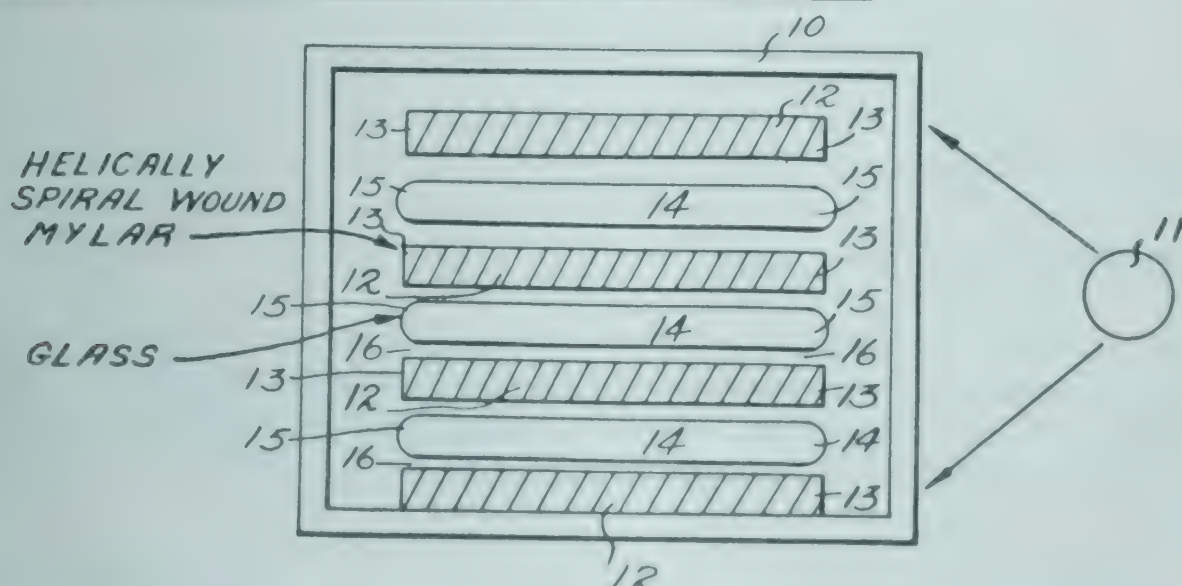
method of accomplishing this sterilization is to rapidly reach the sterilizing temperature throughout the container with electromagnetic energy and then maintain this temperature within the container by keeping these packages for a sufficient time in an environment where the ambient temperature is sufficient for sterilizing, for example 120°C. The materials to be sterilized must contain adequate moisture to allow the generation of steam. The pressure within the vapor-tight container is raised above atmospheric pressure, the temperature above that of boiling water. The higher the temperature and pressure, the shorter the period of time necessary to sterilize.

**Example 1:** About 2 oz. of guinea pig food (Purina) known to inherently contain microorganisms and spores, and having a moisture content of about 7% by weight, was introduced into a glass container measuring one inch in inner diameter and eight inches in length. The container was a glass tube and was sealed at each end so that it was vapor-tight, and placed in a standard microwave oven, and an output energy of about 400 watts was used, the container being spaced in relation to the top, sides and floor of the oven. The oven had an energy capacity of 800 watts. Microwave energy was applied intermittently to the package for 20 minutes at a temperature of 120°C. using about 400 watts energy. After this treatment, the package was opened and the contents were found to be completely sterile. This example was repeated with the temperature maintained above 120°C., notably 120° to 135°C. and 150°C. The higher temperatures permitted a decrease in the time required for sterilization.

**Example 2:** Example 1 was repeated using a vapor-tight package formed of spiral wound Mylar having a thickness of 2 mils. The package was 15 inches long and 0.902 inch in diameter, and sealed at each end. The 800 watt microwave oven of Example 1 was used at the lower setting of 400 watts and the treatment was otherwise the same as in Example 1, the energy being applied for 15 minutes at about 118°C. Ranges of temperature varying between 118° and 121.5°C. were also used in repeated examples. The contents of the package after this treatment were completely sterile.

In Figure 13.4, the numeral 10 indicates the oven to which is applied the electromagnetic energy 11, notably, microwave energy, infrared energy, as explained above.

FIGURE 13.4: STERILIZING FOOD IN SEALED CONTAINERS



Source: J.J. Landy; U.S. Patent 3,215,539; November 2, 1965



The sealed tubes containing the material to be sterilized are supported in spaced relation with respect to the oven and to each other in any suitable manner, as shown at 16. The tubes 12 are formed of spiral helically wound Mylar having sealed ends 13, while the tubes 14 are made of glass having sealed ends 15. The drawing is purely for purposes of illustration. When applying the energy intermittently, as above referred to, it can be applied for from about 2 to 60 seconds at intervals of approximately 10 seconds to 100 seconds, for a total time period to accomplish heating throughout the material and thereby sterilize.

### Sterile Pelleted Food

In conducting certain experiments with laboratory animals, it is important that all food given to the animals in the course of experimentation be free or substantially free of pathogens (i.e., organisms or viruses which may cause disease) in order to insure that disease in the animals has not been caused by pathogens (at least certain specific pathogens) in the animal food. Pelletized food is a most convenient form for animal food.

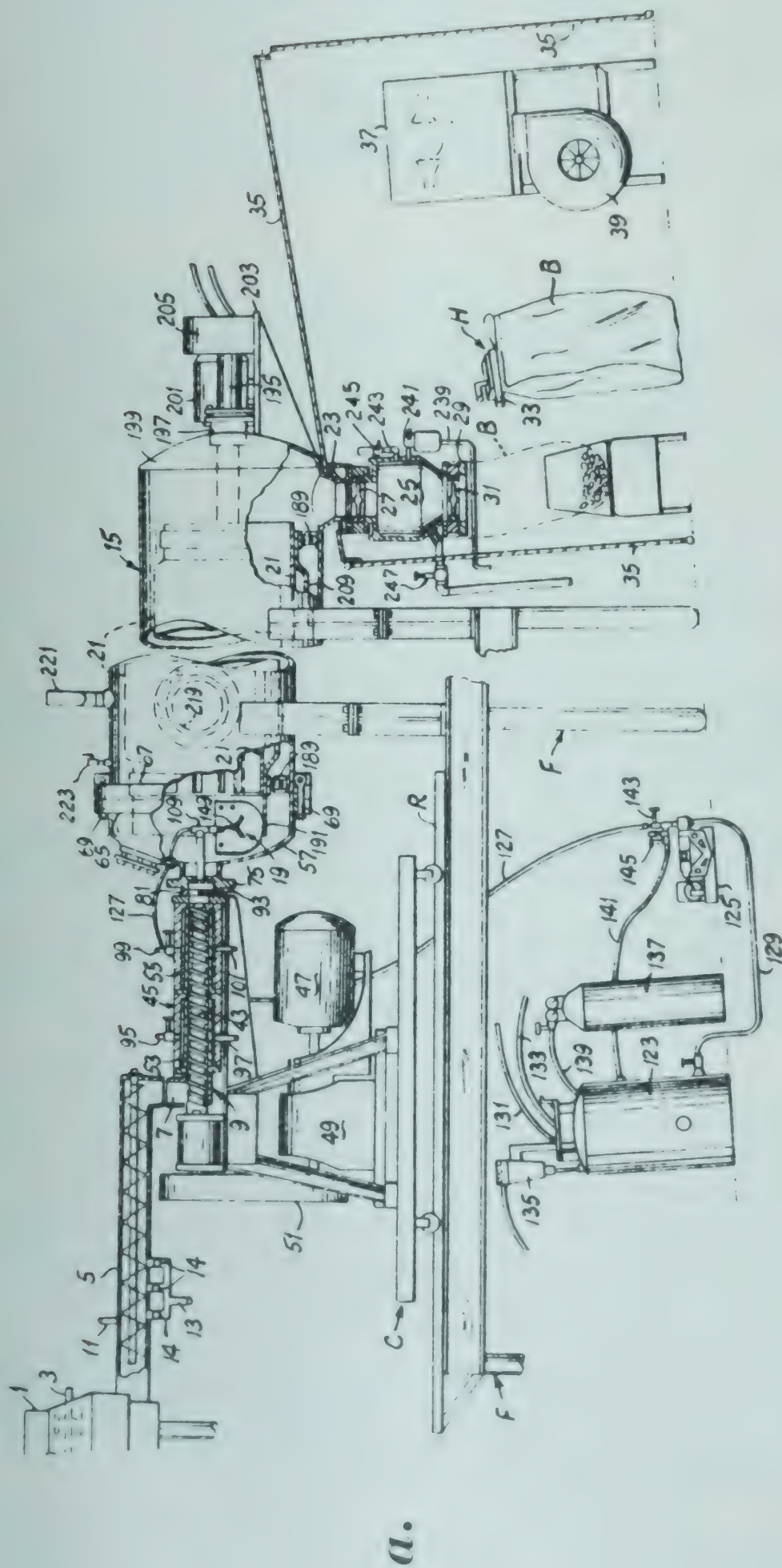
The process of D. Hale, G.S. Vasilakes and R.J. Flier (U.S. Patent 3,268,336; August 23, 1966; assigned to Ralston Purina Company) involves extruding meal with sterilization of the meal during extrusion, pelletizing the extruded sterile meal in a sterile environment, drying the resultant pellets in a sterile environment and packing the dried sterile pellets and sealing the packages in a sterile or aseptic environment. The meal is extruded into a vacuum tank, where the pelletizing and drying occur, and if any pathogens should possibly escape being killed in the extruder, there is a tendency for their cells to explode in the vacuum tank, thereby doubly insuring sterilization.

It is further desirable that the pellets contain fat, also pathogen-free and more particularly to incorporate the fat in the pellets in such manner as to provide for a reasonably uniform distribution of fat throughout the body of each pellet, rather than having the fat concentrated toward the surface of each pellet. This tends to insure that animals will eat all of each pellet, rather than nibbling away only the outside of each pellet, as they would tend to do if the fat were concentrated toward the surface, and thereby avoids waste of food.

Referring to Figures 13.5a, 13.5b and 13.5c and assuming that hopper 1 has been loaded with meal to be formed into pellets, that tank 15 and chamber 25 have been sterilized, that the fat in fat tank 123 and the fat delivery system have been sterilized, and that a vacuum is being maintained in tank 15, operation is as follows. The screw conveyor 5 is driven to feed the meal from the hopper into the extruder 9. Water is injected into the meal in the screw conveyor at 11 to add moisture to the meal. Steam is injected into the meal in the screw conveyor at 13 to bring the temperature of the meal up to 160° to 210°F. for example. The water and steam bring the moisture content of the meal up to 25 to 30%, for example. Screw 43 of extruder 9 is driven to force the meal through port 85. The by-pass valve 89 is initially screwed up to its by-pass position for initially by-passing meal delivered through port 85 down and out through hole 83, until proper heat and pressure are developed in extruder 9 for sterilization of the meal therein.

This is to avoid possible delivery of unsterile meal to extrusion T 109 at the start. Then, valve 89 is backed down to its Figure 13.5b operative position, permitting the meal forced through port 85 to pass through hole 83, port 87, cavity 103 and pipe 105 to the T 109, and

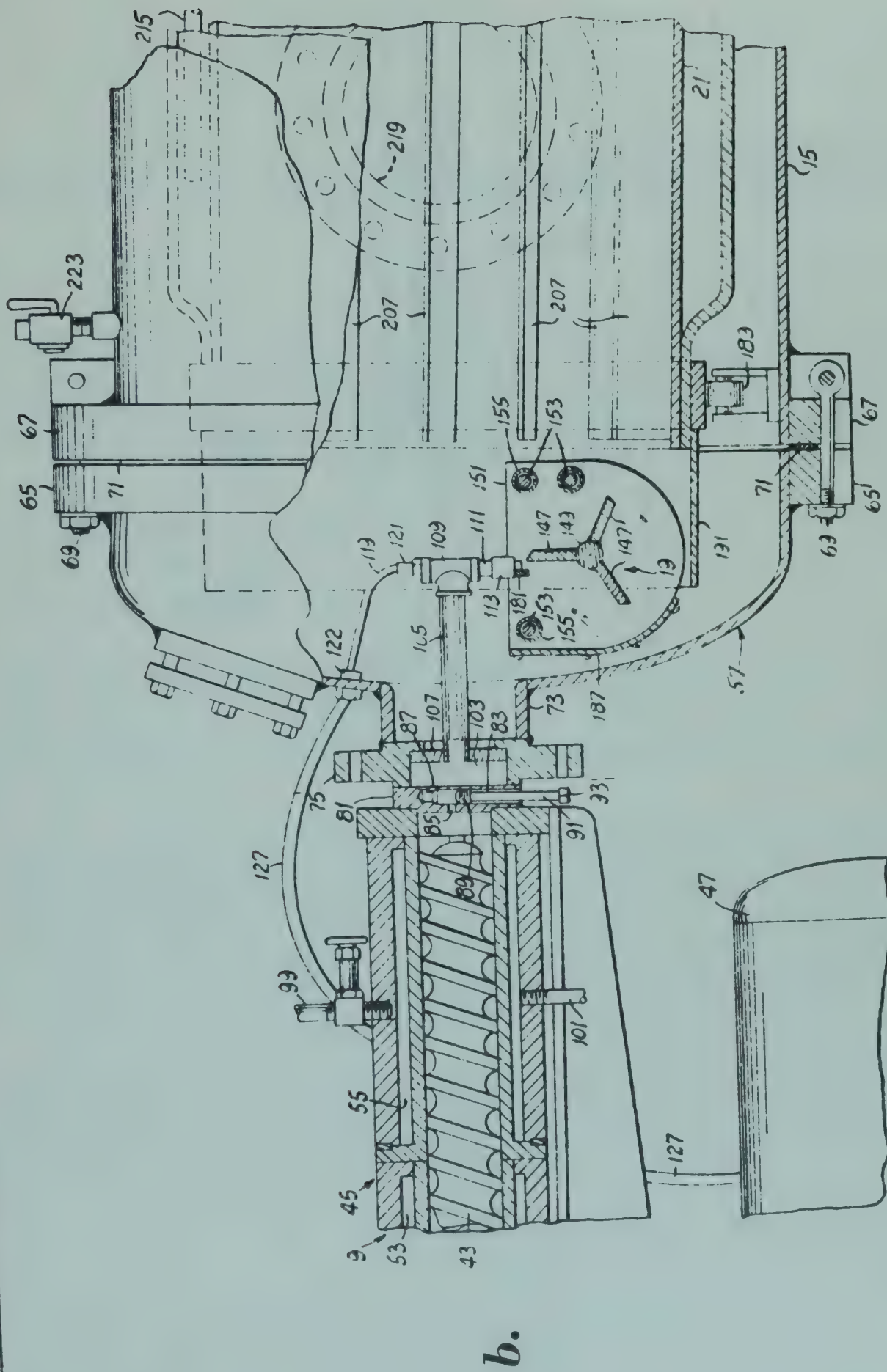
FIGURE 13.5: STERILE PELLETTED FOOD



(continued)

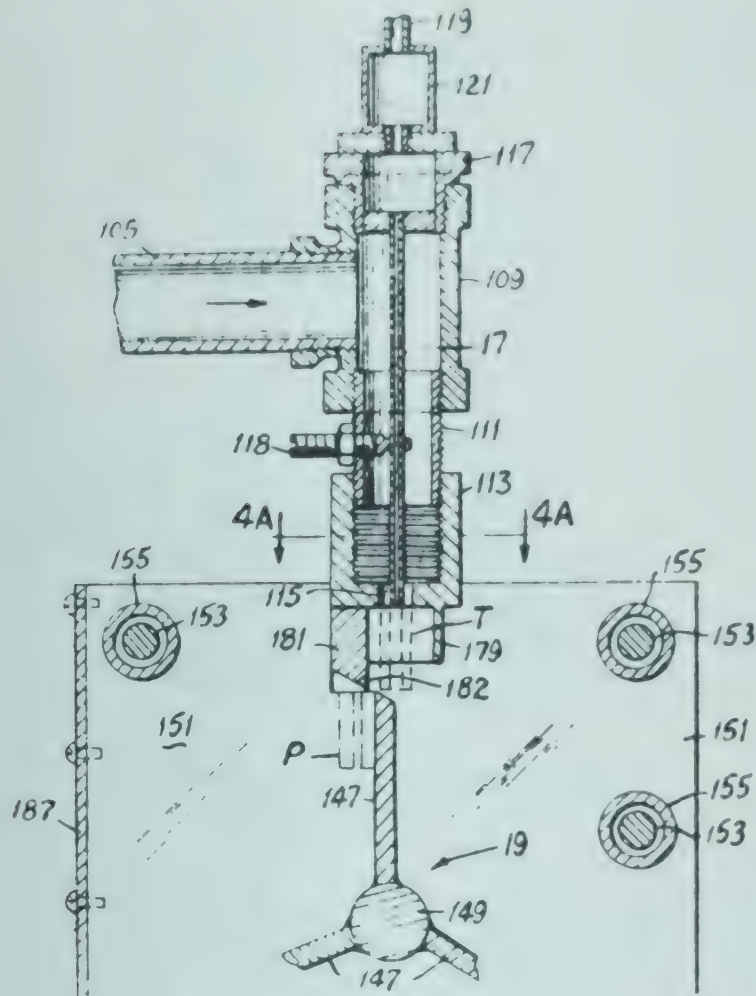


FIGURE 13.5: (continued)



(continued)

FIGURE 13.5: (continued)



Source: D. Hale, G.S. Vasilakes and R.J. Flier; U.S. Patent 3,268,336; August 23, 1966

down through nipple 111 and extrusion nozzle 113. The meal is extruded through rectangular orifice 115 around the lower end of fat injection needle 17 to form the tubular rectangular extrusion T, and fat is delivered internally to extrusion T simultaneously with its exit through the orifice. Since the meal has been sterilized in the extruder, extrusion T is sterile, sterility being maintained by reason of the sterile environment within the vacuum tank 15. If any pathogens escape being killed in the extruder, there is a tendency for their cells to explode as extrusion T exits through orifice 115 into the vacuum in tank 15.

Cutter 19, which is continuously driven, segments extrusion T into pellets P, also within the sterile environment of tank 15. The pellets shot off by the blades 147 of the cutter have their direction reversed by the chute 187 and enter the drying drum 21. They tumble along the drum, becoming dry therein by heat from the steam jacket on the drum, all within the sterile environment of the tank 15, and ultimately fall out of the exit end of the drum. With butterfly valve 27 open, the dried pellets drop into chamber 25.

The operator, in the sterile environment in enclosure 35, and with a charge of pellets in chamber 25, closes valve 27, opens air valve 241 to pressurize chamber 25, pulls the mouth of a bag up around the outlet 29 of chamber 25, then opens valve 31 to blow the pellets from chamber 25 into the bag. Then he quickly closes valve 31, and opens valve 27 for recharging chamber 25 with pellets. The filled bag is then tightly sealed to prevent contamination of the bagged pellets from outside air.



## UTILIZATION OF INDUSTRIAL WASTE AND BY-PRODUCTS

With the increasing demand for processed foods for animals and pets, it is necessary to seek out new sources of economical food. This food must be high in nutritive value and at the same time be appealing and appetizing to the animals which consume it. The processes in this section describe feed compositions prepared from industrial waste and by-products.

### FEED SUPPLEMENTS FROM POULTRY WASTE

#### Offal Processing at 60° F.

A method of processing offal and like by-products and waste products of raw chicken or other fowl which eliminates offensive odors, retains substantially all nutritive values, and adequately preserves the processed material with an extremely low bacteria count is reported by M. Werblud (U.S. Patent 3,032,415; May 1, 1962; assigned to Netex Mink Ranches, Inc.).

The first step in the process is to chill the offal as soon as possible after slaughtering. This does not necessarily require that the temperature be reduced to freezing or below. Instead, it is sufficient if the temperature be reduced from normal body heat to approximately 60° F. One method of achieving this result is to send the offal through a refrigerated screw conveyor. This screw conveyor may be provided with a water jacket through which cold water or a refrigerant passes. The inner wall serves as a heat exchanger and the screw moves the offal along said heat exchanger in order to reduce its temperature to approximately 60° F. The length of the screw conveyor and the speed of rotation of its screw are determined by the single requirement that offal, deposited in the receiving end of the screw conveyor at body temperature, is discharged through the outlet end at a temperature of approximately 60° F. The first step can also be accomplished by icing the product.

The second step is to grind the chilled offal and this is done in motor-driven grinder, which may also be refrigerated and more particularly provided with a water jacket through which cold water or a refrigerant may circulate in order to maintain the material within the grinder at a relatively low temperature. The inner wall of the grinder serves as a heat exchanger between the material being ground and the cold water or refrigerant. It will be understood that the grinding operation takes but a relatively brief period of time and the heat generated thereby would normally but slightly raise the temperature of the material being ground. Such rise in temperature would not ordinarily have any significance in this process and consequently

it would be entirely feasible, although not necessarily preferable, to utilize an unrefrigerated grinder in the place of the refrigerated grinder. The third step in the process is to deliver the ground offal to a mixer and this is done by means of a refrigerated feed screw. This screw has a water jacket through which cold water or a refrigerant may be circulated in order to lower the temperature of the offal or to maintain it at a low temperature. The inner wall of said water jacket serves as a heat exchanger, said inner wall being also the outer wall of the feed screw. The ground offal is maintained at a temperature of between 55° and 60° F. in its passage through the feed screw. Should it happen that the offal enters the feed screw at a temperature higher than 60° F. by reason of the heat generated in the grinding operation, its temperature will be reduced to between 55° and 60° F. in its passage through the feed screw.

The fourth and most important step in the process is the mixing or agitating step in which the ground offal is thoroughly aerated and deodorized. This step takes place in a motorized mixer or agitator having a water jacket in which cold water or a refrigerant may circulate to cool or refrigerate the contents of said mixer. The inner wall of the water jacket, which is also the outer wall of the mixer, is the heat exchanger through which heat is removed from the offal and carried away by the refrigerant. The ground offal is mixed or agitated in said mixer for a period of approximately two hours during which time its temperature is lowered to about 50° to 55° F. All undesirable odors are removed in consequence of this operation and the material is now ready for the next stage in the procedure which is the packing stage.

The ground, deodorized and chilled or refrigerated offal may now be packed in any suitable type of container, for example, corrugated boxes adapted to receive approximately 40 pounds of material. A blast or sharp freeze is now applied to the packaged material in order to reduce its temperature to below freezing. The material will now be preserved until it is used. Raw offal thus processed is highly fit for animal consumption. Its bacteria count is only 31,000 per gram, this being the lowest bacteria count of any animal food prepared from raw offal or the like. It is free of offensive odors and is quite palatable to carnivorous animals such as mink, dogs and cats. The protein analysis of such food exceeds 17% and therefore it possesses good nutritive value.

### Foodstuff from Poultry Offal

An improved process for converting poultry offal (i.e., heads, feet and viscera) into a highly digestible food product is described by I.M. Docken (U.S. Patent 3,071,468; January 1, 1963). The offal material is fed into a hog grinding machine where it is cut and recut.

The ground material discharged from the grinder is then fed into a washer (screened drum revolving through a water bath). From the washer the cleaned ground offal material is pumped into a decanter. The decanter preferred in the practice of the process is known as a horizontal bowl decanter, which has a horizontal bowl which rotates at high speed thereby creating a high centrifugal force which acts in a direction vertical to the axis of rotation to perform rapid and efficient sedimentation of the solids suspended in the liquid. The slurry of offal from the washer is introduced into the bowl through a feed tube in the hollow center shaft. It is then led into the separating area where the high centrifugal force deposits the solids against the wall of the bowl. The liquid, being of lower specific gravity, forms a

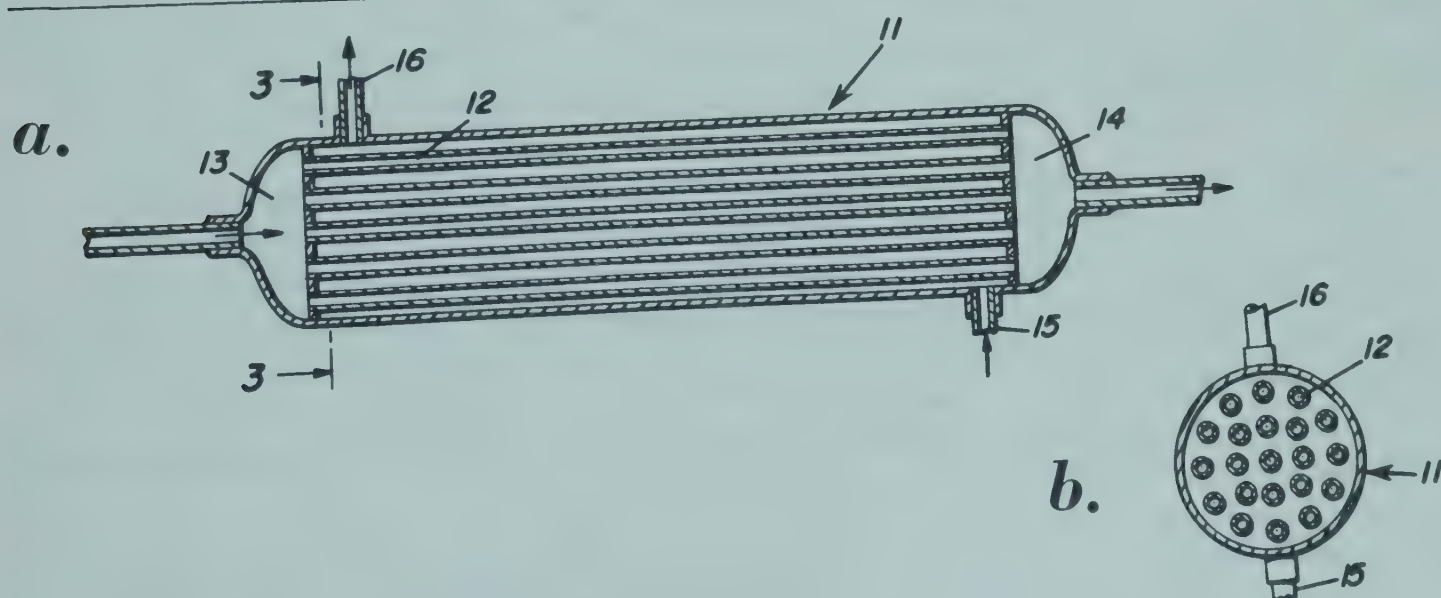


concentric inner layer in the bowl. Inside the rotating bowl is a helical screw conveyor which rotates in the same direction as the bowl but at a slightly lower speed. The flights are designed so that the offal solids which are thrown to the bowl wall are conveyed to one end of the bowl where they are discharged through suitably located openings. The clarified liquid continuously overflows weirs at the opposite end of the bowl. A discharge opening leads the liquid away and keeps it separate from the solids. The decanter, then, will bring the product to the desired water content and eliminate free grease, thus ensuring an end product having uniform moisture, fat and protein content.

From the decanter the material is fed into a vat, to store the offal until it may be admitted into the next step which is a digester. A pump is employed to move the material to the digester. For an understanding of the illustrative type of digester, attention is directed to Figure 14.1 a,b. The digester comprises a large tube or housing 11 through which extend a plurality of smaller conduits 12. Headers 13 and 14 are associated with each end of the smaller conduits. The housing 11 contains a fluid heating medium which surrounds the small conduits. Suitable ingress ports 15 and egress ports 16 are provided in order to effect suitable circulation. The conduits leading therefrom are led to a conventional heat source or heat exchanger. The heating medium must not raise the digester temperature above 140° F., but should maintain it at between 136° F. and 139° F., preferably as near the latter figure as possible.

When the chicken offal is pumped through the digester at the controlled temperature mentioned, the offal reacts autolytically due to the natural enzymes present in the material and becomes a liquefied chicken material, comprising substantially 100% assimilable protein and of a homogeneous thick cream-like consistency. The material is pumped from the vat through the digester and is allowed to circulate until the required amount of digestion or chemical process has taken place.

FIGURE 14.1: TWO VIEW ILLUSTRATION OF A DIGESTER





### Food for Carnivorous Animals from Poultry Viscera

A method and apparatus for converting viscera, such as poultry viscera, into a high quality proteinaceous food for carnivorous animals has been developed by R.H. Moyle and R.A. Moyle (U.S. Patent 3,192,047; June 29, 1965).

Example: This example is explained with reference to Figure 14.2. Crude poultry viscera such as chicken viscera obtained from poultry slaughtering operations may be conveyed in an appropriate manner (not shown) to the grinder 10. For example, the poultry viscera from a conveyor line may be placed in a trough (not shown) containing running water to provide a vehicle for conveying the viscera to grinder 10. Within the grinder 10, the viscera and water mixture is comminuted and forced through a grinder head 50 as discrete solid particles which are desirably less than 1/2 inch and more preferably less than 1/4 inch in length. At this point the comminuted viscera will contain its original content of feces and moreover, because of the crimped nature of the intestines, much of the feces will be trapped in folds of the comminuted intestines.

At the same time, a stream of water from any suitable source (not shown) is charged by a line 52 to the venturi jet 18 for flow therethrough. The pressure normally encountered in civic water lines will be adequate for this purpose when the jet 18 has a discharge diameter of about 1/4" or less. Higher pressure may be used, if desired. As a consequence, a vacuum is induced in the throat or annulus 56 of the venturi means 12. Through the provision of a coupling means 14, such as a hose, between the discharge end of the grinder 10 and the throat section 56 of the venturi 12, a vacuum is formed which is normally sufficient to not only positively pull the comminuted viscera into the throat section 56 but also to expand the comminuted portions of the viscera to substantially completely unfold crimps of the solid portions thereof (e.g., intestines) and hence, expose substantially all of the surface area.

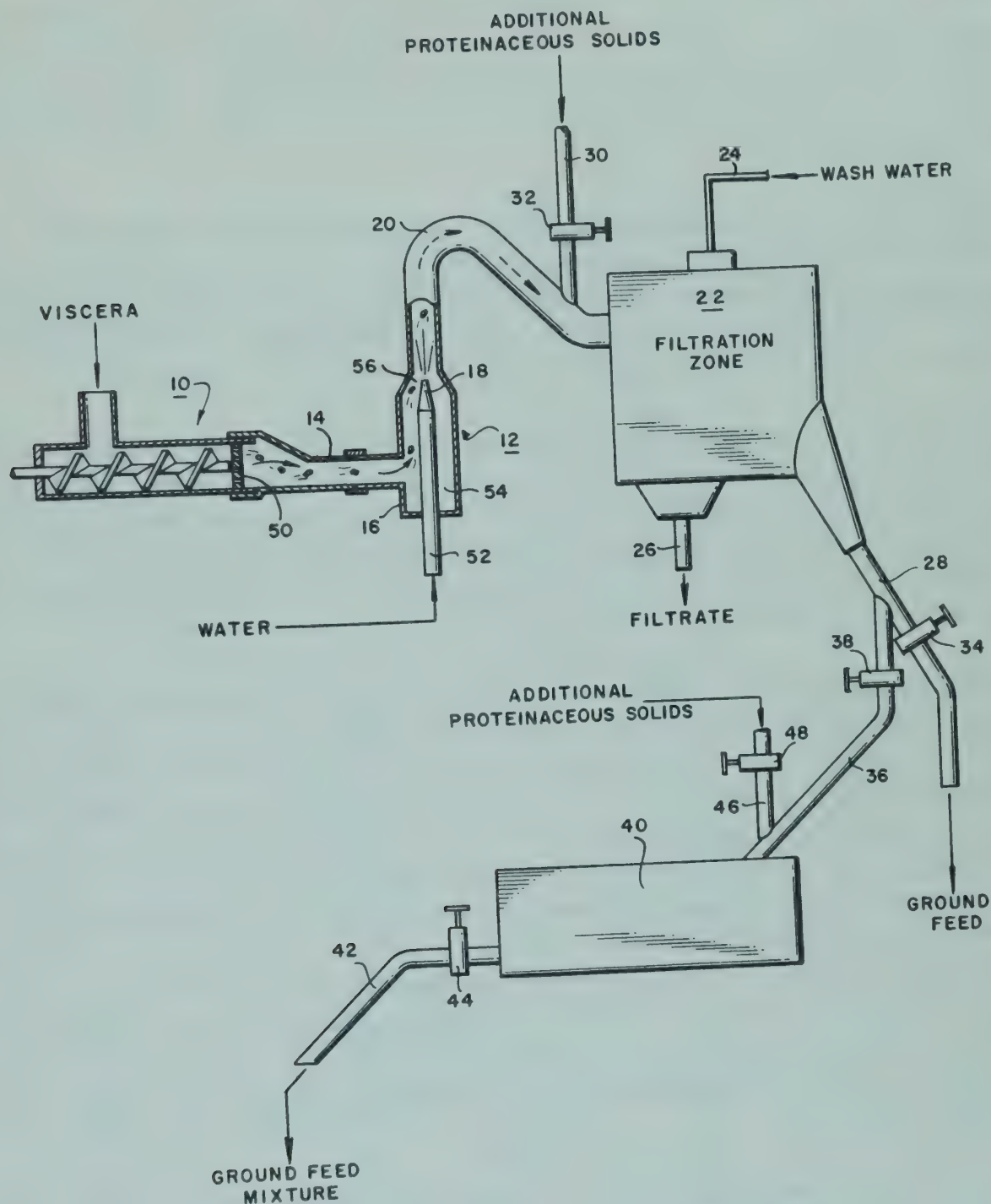
The thus expanded or exfoliated comminuted viscera is induced into the jet of water emanating from the jet 18 of the venturi means 12. Because of the high velocity of water jet, the induced viscera will be subjected to violent turbulent agitation which will act to further shred any shreddable components of the comminuted viscera and literally blast or otherwise remove the minute portions of feces from the uncrimped (exfoliated) comminuted viscera.

The resulting mixture, as indicated, is flowed from venturi means 12 through conduit 20 to a filtration zone 22, wherein water is removed from the proteinaceous solids to thereby obtain a filtrate 26 which will contain in solution substantially all of the feces initially present in the crude viscera. The mixture or slurry in the conduit 20 will suitably contain from about 5 to 50 weight percent of ground particles. The filter-cake that is formed is preferably washed free of any occluded filtrate with water introduced by line 24. The wash water is preferably discarded through line 26 along with the filtrate and is preferably sewered because of its feces content.

The filtration zone 22 is a rotary filter of any desired construction. When it is desired to add additional proteinaceous material to the cleaned comminuted viscera, this may be conveniently accomplished by adding the meat scraps, chicken feet, chicken fat, chicken heads, meals, medicaments, etc., or mixtures thereof, to the filtration zone 22 through conduit 30 together with the slurry from the line 26 whereby the supplementary material



FIGURE 14.2: FOOD FOR CARNIVOROUS ANIMALS FROM POULTRY VISCERA



Source: R.H. Moyle and R.A. Moyle; U.S. Patent 3,192,047; June 29, 1965

will assist in filtration of the cleaned comminuted viscera. The comminuted solid portions of the viscera alone, or in combination with supplementary materials, is discharged from zone 22 by conduit 28 and may be utilized at this point if desired as a food for carnivorous animals. In an alternate method of operation, conduit 28 is blocked at 34 and the solid comminuted material together with added proteinaceous solids is routed by conduit 36 to grinding zone 40, to comminute the added proteinaceous material. Still further quantities of proteinaceous solid may be simultaneously or alternately charged to grinder 40 through conduit 46. The final product discharge from zone 40 will constitute a comminuted substantially feces-free food for carnivores.

### Supplement from Combination of Offal and Feathers

A process by which poultry offal, blood and feathers may be treated simultaneously by the same process to provide an end product which is superior in quality to products resulting solely from the conversion of feathers or solely from the conversion of offal has been developed by P. Speer (U.S. Patent 3,272,632; September 13, 1966). This is described with reference to Figure 14.3.

The wet offal comprising heads, feet and innards are conveyed by suitable means from the slaughtering operation to a grinder or chopper 10. Simultaneously, wet feathers from the slaughtering plant are conveyed to a chopper or grinder 12. If desired, only one grinder may be used for both the offal and feathers. The blood resulting from the slaughtering operation is pumped from the drain trough or collection tank to a conduit 14.

After chopping and dewatering, the offal and feathers together with the blood are deposited in a cooker 16 containing hot oil. The wastes are conveyed through the cooker 16 by suitable means and at a preselected speed until they are deposited on a conveyer 18. The cooker 16 is not a pressure cooker, but rather the pressure within the cooking container is atmospheric pressure. The cooker may be closed for convenience and removal of water vapor, and an exhaust duct 17 may be provided for exhausting vapors to atmosphere.

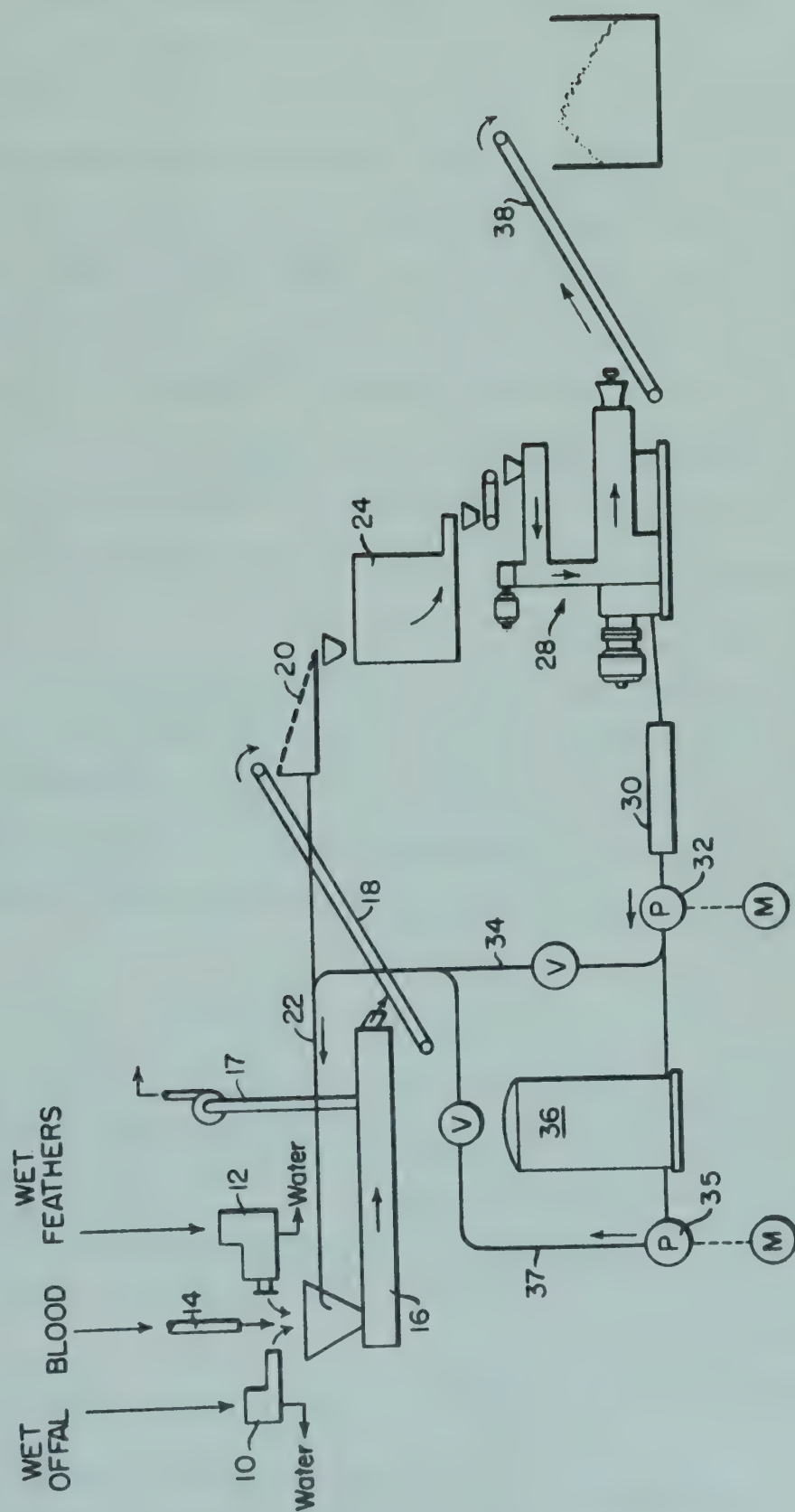
When treating feathers, the temperature of the oil is usually approximately 300° F., but in any event should be in excess of 212° F. in order to provide sterilization where the product is to be used as a feed. The temperature of the oil is determined by the time and temperature relationship necessary to satisfactorily convert the feathers to a product having the desired percentage of digestibility and desired dehydration. It has been found, as an illustration, that where feathers are treated in tallow at a temperature of approximately 300° to 315° F., a time of treatment of approximately fifteen minutes is sufficient to satisfactorily convert the feathers to a usable product. One of the characteristics of this process with respect to the conversion of feathers is that the feathers are "popped" during the hot oil treatment much in the nature of popcorn although the feathers may not be everted in the same manner as popcorn. Where the temperature of the oil is reduced substantially below 300° F. this popping is not so pronounced and although dehydration is complete, the percent of digestibility of the end product is reduced.

The type of oil utilized can be either animal, vegetable or mineral. Where the resultant product is to be used as a food product, the oil utilized should, of course, be edible. Also, the oil should be selected so that the operation temperature of the oil bath is below ignition and decomposition temperature of the oil. The term "oil" as used herein includes materials which are solid, for example, at room temperature but which are liquid at the temperatures of the treatment.

The converted wastes are carried by the conveyor 18 to a fluid screen 20 by means of which readily removable excess oils are drained from the wastes and conveyed by a pipe 22 back to the oil cooker 16. The wastes when reaching the screen 20 may include as much as 50% by volume of oil. The drawing of the oil in the screen should reduce the oil to about 35%. The wastes passed over the fluid screen 20 are deposited in a surge storage bin 24. The wastes are removed from the storage bin 24 by suitable means and deposited on a magnetic



FIGURE 14.3: SUPPLEMENT FROM COMBINATION OF OFFAL AND FEATHERS



Source: P. Speer; U.S. Patent 3,272,632; September 13, 1966

## Utilization of Industrial Waste and By-Products

conveyer 26 for the removal of any ferromagnetic refuse such as wire, staples, etc., which may have come inadvertently mixed with the waste during the slaughtering operation. The wastes are then deposited in a press 28 which removes excess oil and fats from the waste. It is preferred that the oil content be reduced about 22% or less by weight of the product. Where the oil content is higher than 22%, the product is difficult to handle.

The oil content can be controlled by mechanical pressing to about 8% or as low as 3 to 4% by means of solvent extraction. The oil content may vary depending on the desired end use of the product. In the case where the product is to be used as a protein feed, the oil content may be reduced to approximately 8 to 15% which is a common fat analysis range for feed. The oils and fats removed from the wastes are deposited in an oil sump 30 and conveyed by a pump 32 through a conduit 34 back to the conduit 22 leading to the oil cooker. Where the rate of oil and fat removal by the press is in excess of that necessary to provide makeup oil in the oil cooker, the surplus oils are conveyed to an oil storage tank 36. The tank 36 is preferably heated to preheat the oil for subsequent use in the cooker.

The oil in the tank 36 is removed therefrom by a pump 35 through a conduit 37. Suitable valving is provided in the conduits 34 to 37 to achieve the desired adjustment in the return flow of oil to the cooker. Where the poultry process is high in waste fat, the process will be more than self-sustaining with regard to the oil used in the cooker, which enhances the economics of the process.

The wastes are discharged from the press onto a conveyer 38 leading either to storage or the dry end product or, if desired, to suitable means for packaging the product. A moisture content at the end of the process should be 15% or less by weight of the product, no greater than 8% in the case of protein feed. In any event, the moisture content should be sufficiently low to eliminate souring of the product during storage and to render the product easy to handle. The desired dehydration of the waste occurs in the oil cooking step and only minor amounts of moisture, if any, are removed in the press 28.

The digestibility of the end product is particularly good; for example, when a combination of offal and feathers in the approximate proportions normally found in poultry were processed as described above in tallow for fifteen minutes, at a temperature of approximately 315°F., the digestibility of the end product prior to final processing was approximately 82.7% with protein 60.78%, fat 31.05% and moisture 0.34%. The digestibility was determined by a conventional pepsin-hydrochloric acid test. This percentage digestibility indicates a high percentage of protein available in the end product which makes it particularly attractive for use as animal feed or a fertilizer. In this connection, it is believed that the digestibility should normally be at least about 70%.

### Low Ash-Content Supplement from Poultry Bones

W. Kuster (U.S. Patent 3,370,954; February 27, 1968; assigned to Eagle-Ottawa Leather Company) has developed a method suited to the processing of poultry by-products containing leg bones and neck bones, for example, and to the processing of other high bone content materials. Heretofore such by-products have not been used because of the undesirably high ash content of the final product. By employing this process, the presence of bones, etc. presents no problem and the high ash content is eliminated as described.



## Utilization of Industrial Waste and By-Products

The selected animal by-products are deposited continuously or in batches in a hydrolysis chamber and are subjected to hydrolysis with phosphoric acid in aqueous solution at a pH of preferably less than about 2. Hydrolysis of the protein in the animal by-products is promoted by the application of heat such as by injecting steam into the solution or heating by coils or jackets. Suitably, the heat is regulated so that the reaction temperature is under 300° F. and more preferably less than about 250° F. so that the protein is not adversely affected nutritionally.

Hydrolysis of the protein is continued until the protein reaches approximately the polypeptide stage. (It should be understood that the protein and protein derivatives at this point are not all in a homogeneous stage, but the term "polypeptide" is intended to cover the average condition of the protein material. Thus some of the protein may be in the form of a peptone and just soluble in water while some of the protein may be converted to the peptide stage.) When the protein has been converted to the polypeptide state, it is removed in aqueous phase from the hydrolysis chamber. Portions of the starting animal by-product protein that have not been so converted may be left in the hydrolysis chamber for further hydrolytic treatment. If the liquified protein that is removed from the hydrolysis chamber contains undesired solid portions such as hair of the animal by-products, these may be separated therefrom as by filtering or screening.

During the hydrolysis treatment the fat portions of the animal by-products, by reason of the heat applied, become fluid and liquified. During the removal of the liquified protein and during any screening or filtration which may be employed, the liquified fat is generally obtained along with the aqueous protein phase. In a continuous process, and especially where a great deal of agitation occurs during the hydrolysis step, the protein and liquified fat continuously being removed from the hydrolysis chamber may be in emulsified condition. Alternatively, and particularly in a batch process where a hydrolysis is executed slowly and without much agitation, a majority of the fat may separate on top of the aqueous phase and can be easily removed. Where such separation does not occur, or where an appreciable emulsification has occurred, separation of the fat may be accomplished by any conventional technique.

The amount of fat removed will depend upon the ultimate consistency of the product desired. When the product is to have a paste consistency, sufficient fat should be left so that the desired homogeneous paste can be obtained. In any event, the fat level should be adjusted so that it will permit obtaining the desired consistency in the final product. The separated polypeptide aqueous phase protein which may contain a preselected desired amount of fat is then treated with the selected calcium compound.

Calcium carbonate, for example, is added to the solution to bring the pH up to about 5 to 7. Insoluble phosphate salts are thereby produced. The precipitated salts are conveniently separated by any suitable technique such as filtering or centrifuging and the remaining solution treated as desired to obtain the final product. Where a paste is desired, the water content is reduced as by evaporation until the paste consistency is produced.

Where a dry product is desired and a higher ash content can be tolerated, drying is carried as far as possible before the material becomes too tacky for handling. At that point sufficient phosphoric acid and calcium carbonate are added to tie up enough of the remaining



water as water of hydration so that the product is rendered dry (commercially dry) and free-flowing. If all of the phosphoric acid initially added for hydrolysis is not neutralized in the precipitation step, the addition of calcium carbonate alone at this point may be required to accomplish the intended purpose.

### PRODUCTS FROM MEAT INDUSTRY

#### Blood Meal from Hydrolysis of Whole Blood

Since large quantities of blood meal are annually available in the United States (about 160,000 tons) and since blood meal contains about 75% protein and is an excellent source of lysine and tryptophane, nutritionists have attempted for many years to utilize this material in animal rations. However, relatively little blood meal has been used in animal feeds, due in part to various investigations indicating the unpalatability, poor digestibility and low biological value of the commercially available blood meal.

A process for treating whole animal blood to improve its availability in animal nutrition is described by H. Gershon (U.S. Patent 2,996,383; August 15, 1961; assigned to Pfister Chemical Works, Inc.). The whole blood is subjected to moist heat such as is available in the common autoclave type of pressure cooker, for periods ranging from about one-half to two hours under pressures of from about 12.5 to 22.5 psig. The fresh blood may be treated in the above fashion directly by the meat packer, for example, or if desired, a subsequent processor may autoclave the commercial dried blood meal after first slurring it in water. Preferably, the whole blood is autoclaved for about an hour while maintaining the pressure at 15 psig.

Subsequently the treated blood is dried, preferably at about 70°C. Thereafter the material is ground and a treated blood meal, according to this process, results. It is believed that the heat treatment partially hydrolyzes the proteinaceous content of the blood, effecting vast improvement in the digestibility and biological value of the processed blood meal protein supplement and the feeds containing the same.

The nutritive values and feed efficiencies of the untreated blood meal are surpassed by feeds containing whole blood treated by this process in which blood has been subjected to a moist heat treatment at pressures ranging from about 12.5 to 22.5 psig. The maximum nutritive values and feed efficiencies occur when the blood is autoclaved at 15 psig. Five samples, each containing 65 parts by weight, of the basal ration were provided. To these samples 35 parts of the protein supplements indicated in the table below were isonitrogenously added to produce mixed feeds and the feeds fed to duplicate groups of chicks, Nichol's cross 180 cockerels, for 4 weeks. The various protein supplements utilized and the feed efficiencies (gain in weight per amount of feed consumed, G/F) were as follows:

| <u>Protein Supplement in Feed</u>           | <u>G/F</u> |
|---|------------|
| 100% soy bean oil meal                      | 0.48       |
| 95% soy bean oil meal, 5% B <sub>8</sub> *  | 0.52       |
| 85% soy bean oil meal, 15% B <sub>8</sub> * | 0.49       |

(continued)



(continued)

| <u>Protein Supplement in Feed</u>           | <u>G/F</u> |
|---|------------|
| 75% soy bean oil meal, 25% B <sub>g</sub> * | 0.46       |
| 50% soy bean oil meal, 50% B <sub>g</sub> * | 0.43       |
| 100% B <sub>g</sub> *                       | 0.33       |

\* B<sub>g</sub> = 50% autoclaved blood meal, 25% hydrolyzed feather meal, 25% hoof meal.

Thus, the feed efficiency of soy bean oil feeds is increased by the substitution of up to about 20% of the soy bean oil meal, by blends of heat treated blood meal and keratin. Additional growth response is obtained by feeding as a protein supplement 50% treated blood meal plus 50% keratin (hydrolyzed feather meal).

#### Blood Meal from Acid Hydrolysis of Whole Blood

E.H. Hess (U.S. Patent 3,352,685; November 14, 1967; assigned to Victor F. Weaver, Inc.) has a process in which acids, either alone or in combination with sequestering agents are added during the coagulation of the whole blood to influence the formation of a granular solid which is readily separable from the supernatant liquid in almost quantitative yields by such economical means as filter pressing and centrifugation.

Example 1 — Chicken blood: Four samples of whole chicken blood taken from the same previously homogenized batch and of equal weight were provided. No acid was added to samples 1 and 2. The pH of samples 3 and 4 was adjusted to 6.1 by the addition of 2.5 parts of orthophosphoric acid per 1,000 parts liquid blood. To samples 2 and 4 were added 1.0 part of N-(hydroxyethyl) ethylene diamine triacetic acid. All samples were autoclaved at 15 psig for 15 minutes. Thereafter the coagulum in each of the autoclaved samples was subjected to a filter press treatment of 150 psig and the amount of supernatant released from each was measured.

Moisture determinations were made on a part of the press cake from each treatment and "rate of drying" studies were made on another portion by allowing them to air dry for 24 hours at room temperature. These later portions when dry were in turn used to measure the comparative in vitro protein digestibilities using the enzyme pepsin in dilute acid medium. The essential detail of this procedure is offered in Methods of Analysis, Association of Official Agricultural Chemists, Ninth ed., p. 286. In the table below are listed the results of this experiment.

|  | Sample 1<br>No<br>chemical<br>additive | Sample 2<br>Seques-<br>tering<br>agent<br>only | Sample 3<br>Acid only | Sample 4<br>Acid plus<br>seques-<br>tering<br>agent |
|--|--|--|-----------------------|---|
| Percent total H <sub>2</sub> O<br>removed by filter-<br>press..... | 70                                     | 68   | 78                    | 78  |
| Moisture content after<br>24 hr. air-drying.....                   | 22.4                                   | 18.0   | 9.8                   | 10.4  |
| Percent total protein<br>recovery.....                             | 99                                     | 99   | 100                   | 98  |
| Percent pepsin<br>digestibility <sup>1</sup> .....                 | 98                                     | 91   | 92                    | 94  |

<sup>1</sup> Commercial meal — 50% digestible under conditions of test.

Example 2 — Improved digestibility as determined by chick feeding experiment: Quantities of chicken blood meal were prepared according to the techniques described in Example 1 (Sample 1—no chemical additive, Sample 3—acid only, and Sample 4—acid plus sequestering agent). These samples along with a quantity of commercially dried chicken blood meal were assayed for total crude protein and incorporated as the sole sources of protein into a series of test diets as set forth by Scott. (Hinners, S.W.; Scott, H.M.: A Bioassay for Determining the Nutritional Adequacy of Protein Supplements for Chick Growth, Poultry Science 39:176, 1960.) All test diets were identical in composition (dextrose, corn oil, vitamins, minerals, etc.) except for the type of blood meal used. Each was added so as to provide 13% crude protein in the total chick diet.

From a group of 100 one week old cockerels were selected 64 of most uniform weight and these in turn were divided into 8 groups of 8 chicks each, each group being placed in its separate compartment in a standard chick battery. Duplicate lots were fed their respective diets for a period of three weeks during which time feed consumption was measured and individual chick weight gains were periodically determined. At the end of the test period all data were calculated, and subjected to rigid statistical analyses. In the table below are listed summary data:

| Diet   | Com-<br>mercial<br>meal | Sample 1<br>No<br>Chem.<br>Additive | Sample 3<br>Acid<br>Only | Sample 4<br>Acid-se-<br>quester-<br>ing agent |
|--|-------------------------|-------------------------------------|--------------------------|---|
| Average wt. gain (in<br>grams) per chick during<br>3 wk. period..... | 77                      | 128                                 | 123                      | 128   |
| Feed consumed/weight<br>gained.....                                  | 4.50                    | 3.35                                | 3.30                     | 3.15  |

These data clearly demonstrate the outstanding superiority in nutritive value of the three experimental meals as compared to a commercial product. They further demonstrate that the use of acid to produce a more easily filterable coagulum has no adverse effect on nutritive value and that even in the relatively air free autoclave a small amount of sequestrant is of value in preventing metal catalyzed oxidation, thus preserving nutritive value.

### Economical Blood Meal Process

An economical means of producing blood meal is described in the process of P. Filstrup (U.S. Patent 3,450,537; June 17, 1969; assigned to Aktiebolaget Separator, Sweden). The raw blood is continuously led through a heating device in which the blood is heated to such a high temperature that it coagulates, the coagulated blood is dewatered in a continuously operating dewatering device, and the coagulated and dewatered blood is finally dried in a continuously operating drying device. The heating of the blood above its coagulation temperature can advantageously be achieved by direct injection of steam. Further, the raw blood, before being heated in the heating device, is preferably preheated to a temperature below the coagulation temperature, as this has proved to have a favorable effect upon the dewatering step subsequent to the heating and coagulation.

The apparatus comprises a combination of elements connected in series, namely, a raw blood container, a pump, a steam-heater, a dewatering device and a drier. The raw blood container is preferably provided with a preheating means. The dewatering device which is preferably a continuously operating, horizontal sludge centrifuge (a so-called desludger), separates about 2/3 of the water quantity which is to be eliminated in order to obtain a



final product with a water content of 5 to 10%. The blood mass, dewatered, is fed to a conventional drying device of the continuously operating type in which it is dried to a final water content of 5 to 10%.

### Feed Product from Animal Manure

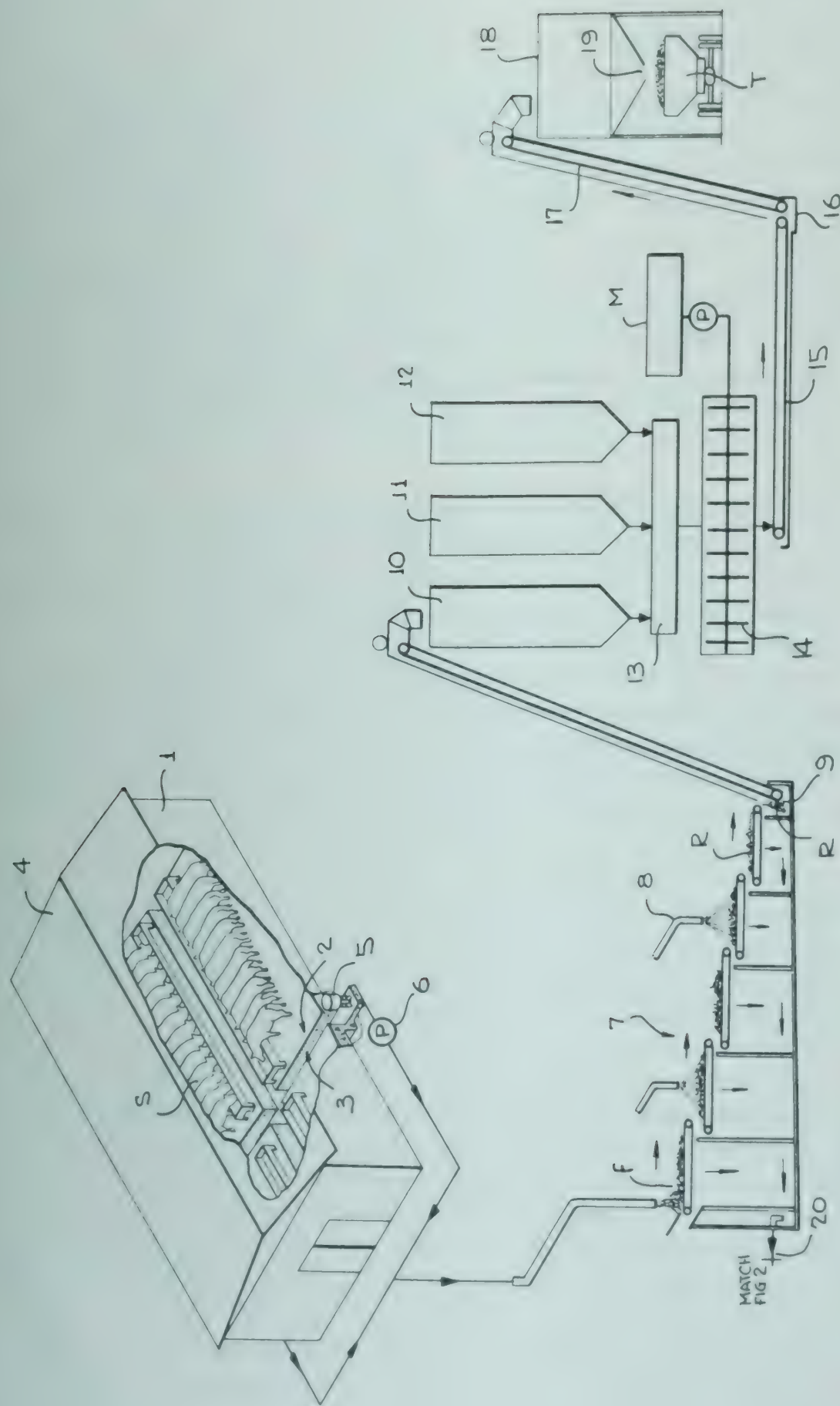
Animal manure is used as a source of nutrients and feed in the process developed by W.B. Anthony (U.S. Patent 3,375,116; March 26, 1968). As shown in Figure 14.4, there is disclosed a barn 1 or other confining means, which has sloping concrete floors 2. In the floor structure, there is provided collection pits 3 for collecting fecal matter from the confined animals. Steers 5 are indicated as being confined. Dairy herds would normally be provided loose housing with conventional comfort stalls. The floors of the enclosure are preferably washed daily by flushing or hosing with water. The floor should slope toward the aforesaid collection pits 3. The collection pits 3 are provided with an agitator 5 which, of course, when in use, is inside the barn and preferably in the pit. The agitator forms a slurry of the water and the manure and a pump 6 conducts the slurry to vibrating washer screen means 7.

The vibrating screen means 7 performs its usual dewatering function. Where limited water is used in making the initial slurry, it will usually be desirable to carry out further washing of the manure by means of spray pipes 8 and this is accomplished while the manure passes over the vibrating screens which, as illustrated in Figure 14.4, are in stepped relationship. The coarse fibrous water residue R is deposited at 9 in the screen means and from there this water residue is conveyed to a suitable holding bin 10. A grain holding bin 11, and a feed supplement holding bin 12 are provided and all of the said bins discharged into common manifold type weight hoppers 13 and there the said wet residue grain and supplement are commingled and from the said weight hoppers the commingled material is conducted to a mixer 14 and to the mixture in the mixer, molasses from a source M, may be added if desired.

The mixture from the mixer is discharged onto a conveyor 15 to a suitable trough-like member 16 and from this trough-like member 16 the mixture is conveyed by an elevator or similar device 17 into a storage discharge bin 18, which bin has a controlled outlet 19 for releasing the said mixture into a truck T or the like. The mixture of the wet residue with the grain and feed supplement added thereto is a complete feed for cattle.

The water that is utilized in watering the manure on the screens, it has been found, contains valuable vitamins, bacteria and protozoa and microbial residue which represents a valuable source of protein. Therefore, it is important that these valuable constituents be not wasted. In a process for the fecal wash water, the solids as well as dissolved nutrients may be concentrated by evaporation. The concentrated wash water would contain much microbial residue, fine particle feed residue, B-vitamin and other undefined biological and organic residue. This product has value as a feed supplement for various classes of animals. It is also valuable for inclusion in medium use for commercial growth of micro-organisms. Although the product can be used in a semi-solid state, the dried material has wider use than this. In commercial operations the product would need to be blended with other feed ingredients to produce a standard product. The following tests were made on three steers to show the efficacy of the feed supplement: Yearling steers were confined on concrete and fed a high grain feed mixture. The manure was collected daily and thoroughly mixed with

FIGURE 14.4: FEED PRODUCT FROM ANIMAL MANURE



Source: W. B. Anthony; U.S. Patent 3,375,116; March 26, 1968



## Utilization of Industrial Waste and By-Products

water. Solid material was allowed to settle and the aqueous layer was poured off. The water washing was repeated and the fecal residue remaining was stored at approximately 33° F. until needed for feeding. The wet fecal residue was mixed with the basal feed in the ratio of 40 parts of wet residue to 60 parts of basal feed. Feed was mixed thoroughly and at the time of mixing, dried yeast was added at the rate of 1 pound per 100 pounds of feed. The final mixture was held in burlap bags about 12 hours before feeding.

Three yearling steers were used in the feeding test. These cattle performed satisfactorily on a steer fattening feed for 7 weeks prior to the time they were placed on test. A 14 day preliminary feeding period on the test ration preceded the experimental period. No difficulty was experienced in getting the cattle to consume the experimental feed. No outward symptoms of harm resulted to the cattle as a consequence of consuming the mixture containing fecal residue. Daily live weight gain and feed efficiency of the cattle were excellent for steers of this weight, age and condition. Cattle fed the fecal residue mixture gained over 3 lbs. daily and required less than 700 lbs. of dry matter per 100 lbs. of gain.

### Protein, Fat and Bone Meal from Dry Rendered Tankage

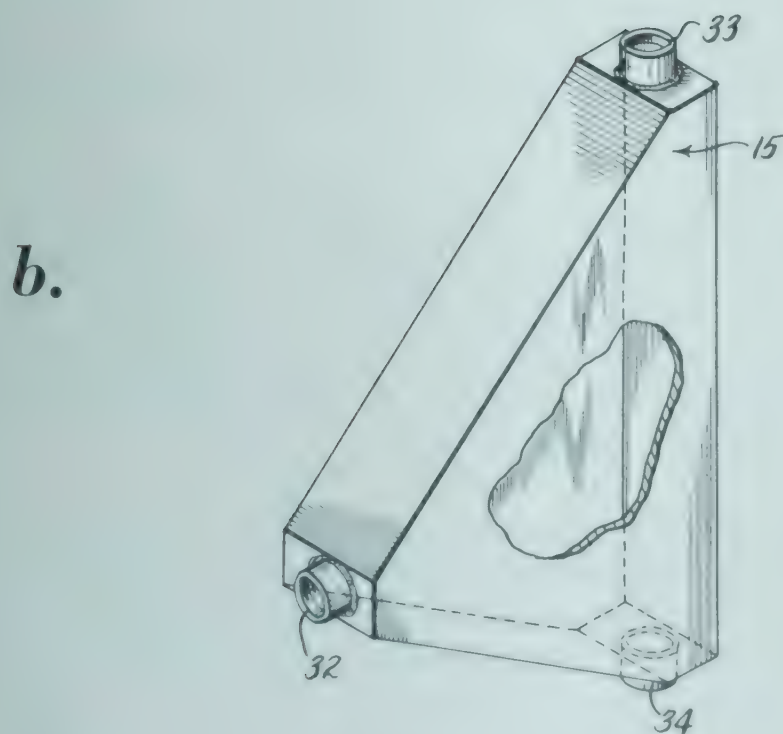
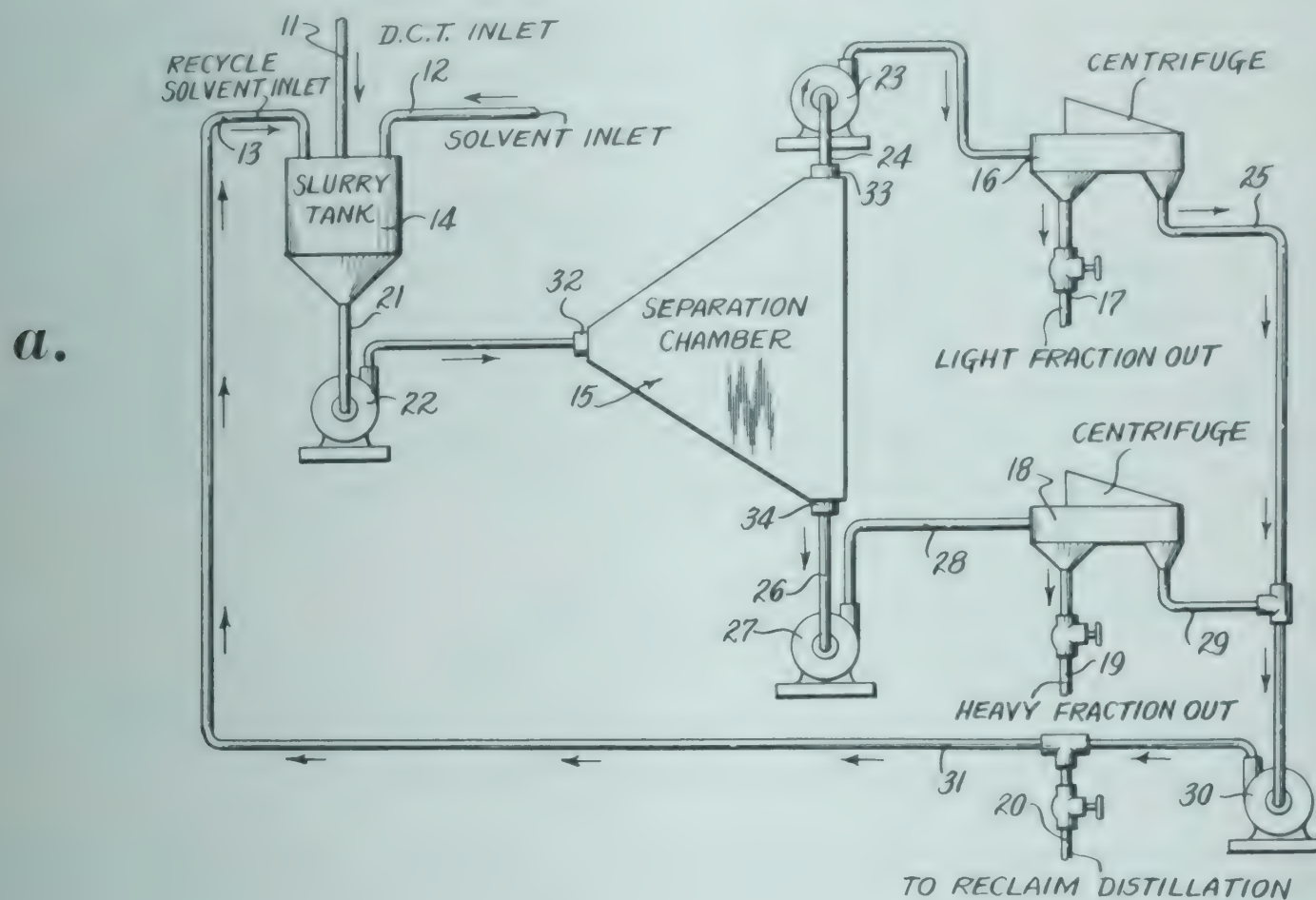
The separation of the proteinaceous material, the bone and the fat into three separate constituents from dry rendered tankage is described by J.E. Thompson (U.S. Patent 3,099,561; July 30, 1963) with reference to Figure 14.5 a and b.

When fat is rendered the solid material that separates therefrom is removed, and comprises a light portion and a heavy portion, as well as the retained fat adhering thereto. A typical analysis of such material as obtained from a sample of cracklings produced by a meat packer, comprises a solid material containing approximately 25% bone meal, 55% protein, 10% fat and 10% moisture. On solvent extraction the fat may be removed and would include any component which is extractable by the solvent used, that is, color bodies, mineral oil, etc. The light solids, mostly dried soft tissue would be high in protein when obtained from good quality dry rendered tankage.

It would also include any light contaminant or adulterant that might be in the raw material, that is hair or sawdust. The heavy solids, mostly bone, when obtained from good dry rendered tankage would include contaminants or adulterants of high density, such as sand, glass, metal scraps, etc. The dry rendered tankage is first run through a hammer mill or treated by other methods for subdividing material so that it will give a product that will pass through a 10 mesh screen and be retained on a 20 mesh screen.

The dry rendered tankage is introduced through the conduit 11 and a solvent, such as carbon tetrachloride is introduced through the conduit 12, both of these admitted through respective materials to the slurry tank 14. The slurry from the bottom of this tank is passed by means of the conduit 21 through the pump 22 to the separating chamber 15 and is admitted at inlet port 32 at the side of this chamber. This chamber allows the flow of the material across the chamber in an even manner where the flow of the material decreases as it progresses from the inlet in the chamber. The light material, protein, rises to the top of this chamber and is removed by the pump 23 through the conduit 24 and delivers the slurry of the light material and a solvent to the centrifuge 16, where the solvent is removed from the light constituent,

FIGURE 14.5: PROTEIN, FAT AND BONE MEAL FROM DRY RENDERED TANKAGE





the solvent being removed from the centrifuge by the conduit 25 and the light constituent through the conduit 17. The heavy constituent, bone meal, is removed from the bottom of the separation chamber 15 by means of the conduit 26 passing through the pump 27 and is admitted by means of the conduit 28 to the centrifuge 18, where the heavy constituents are removed from the centrifuge by means of the conduit 19 and the solvent is removed from the centrifuge by means of the conduit 29. The flow of the solvent in the conduits 25 and 29 is controlled by means of the pump 30 operating through the conduit 13 into the slurry tank 14.

A certain portion of the solvent and its dissolved fatty material is removed from the conduit 31 by means of the valve 20 and passed to a distillation unit (not shown) where the solvent is separated from the fat, which solvent, after separation is readmitted to the process through the conduit 12. The separation chamber referred to previously is shown in detail in Figure 14.5b and is generally triangular in shape. The solvent and the suspended material is admitted on the side through the opening 32 and flows across the chamber to the outlets 33 and 34 therefrom, the outlet 33 being at the top allows the lighter material floating with the solvent to be removed therefrom and the outlet 34 being at the bottom allows the heavy constituent admixed with the solvent to be removed from the bottom at this point.

It is readily seen that the rate of flow of the solvent within the chamber decreases progressively as the material moves across the chamber thereby allowing the lighter constituent and the heavy constituent to separate within the chamber due to gravity separation. The solvent used in this modification of the process is intermediate in density between the heavy constituent and the light constituent and thereby produces a sink-float type of separation, while the fatty constituents are dissolved in the solvent. The solvent referred to in the example is carbon tetrachloride, but any solvent having an intermediate density may be used, such as chloroform, perchloroethylene, tetrachloroethylene, trichloroethane, tetrachloroethane, trichloroethylene and ethyl chloride.

### Feed Supplement from Tannery Fleshings

A method of producing a nutritional feed supplement rich in protein and fats for addition to animal feeds from tannery fleshings and trim has been developed by W. Kuster (U.S. Patent 3,000,742; September 19, 1961; assigned to Cal-Tan Research Products Corporation). The raw materials with which this process is employed consist of green fleshings, limed fleshings, limed hide trim and green hide trim. Preferably the limed fleshings and limed hide trim are handled together. These materials initially have a pH in excess of 9 and as high as 12 or more and accordingly are delimed in a tank with strong hydrochloric acid or some other suitable acid which neutralizes the lime to form a water soluble salt such as calcium chloride. The acid treatment is continued until the interior of the largest pieces no longer shows red to phenolphthalein and indicates that the calcium hydroxide originally present has been almost entirely converted to the very water soluble calcium chloride. This calcium chloride can now be removed by simple water washing.

The total chloride content of the wash water is checked so that it is about equal to that of the incoming wash water from the water supply. After this treatment the limed fleshings and trim may have a pH of about 7 or lower in the ambient fluid. The interior of the pieces of hide, fleshing and trim are preferably brought down to a pH lower than 8. This indicates



that the calcium hydroxide has reacted with the acid to approach neutrality. Preliminary pretreatment of the green trim is separately conducted in the same or a similar vessel (this may be a revolving drum). As a first step the green trim is first agitated with lime and sulfides in order to remove the hair, then in the same vessel the lime and sulfides are removed by acid treatment. Hydrochloric acid and water are added and this results in the formation of water soluble salts and the hydrogen sulfide passes out of the solution as a gas.

Preliminary treatment of green fleshings is not usually required. Both the green and limed fleshings and trim are deposited continuously or in batches in the hydrolysis chamber and thereafter the process is continuous. The hydrolysate in the chamber is maintained at a pH of between 1 and 2.5 by addition of an acid such as hydrochloric either by automatic metering equipment or manually. Undue quantities of salt are avoided by maintaining as high concentrations of material as possible and as high a pH as possible consistent with good hydrolysis. Live steam is also preferably injected into the hydrolysis chamber to raise the temperature thereof and also to thoroughly agitate the ingredients and insure that the newly introduced unhydrolyzed material is kept submerged and in the dilute acid and is prevented from floating in or adjoining the upper fat layer in the chamber to insure continued hydrolysis.

The temperature in the hydrolysis chamber is determined by the boiling point of the mixture in this chamber and normally is slightly above the boiling point of water at atmospheric pressure. This prevents damage to the fats which would occur at higher temperatures and maintains their digestibility, which is very desirable from a nutritional standpoint. A temperature slightly above 212°F. is a minimum, and the maximum temperature should not go above 250°F., if the digestibility of the fat is to be maintained at its optimum. It is possible to hydrolyze at temperatures higher or lower than the boiling point of water at atmospheric pressure. The more concentrated the material entering the hydrolyzing chamber, the higher the permissible temperature and the less concentrated the material, the lower the temperature.

Instead of live steam, a steam jacket or steam coil may be employed in the hydrolyzing vessel, then higher concentrations could be employed and a higher temperature would result. During hydrolysis the protein molecules absorb one or more water molecules and this assists in breaking down the complex protein molecules to their degradation products such as polypeptides and in some cases amino acids. Substantially all the remaining hydrogen sulfide boils off during this step. Hydrolysis continues for a period of about ten to twenty minutes, depending upon the condition of the raw material and on the acid concentration.

Ultimately, if hydrolysis were continued, the formation of amino acids and finally ammonia would occur. However, a complete destruction is not carried out. The hydrolysis is arrested at approximately the polypeptide stage so that the proteins have become water soluble. The fat, by reason of the heat, has become fluid and hence the resulting mixture of fat and protein derivatives being all in a fluid state is capable of being screened to remove hair and other impurities from the liquified material. After being screened by a coarse grating located at the discharge of the hydrolysis chamber, the material enters one or more digesters where hydrolysis is continued, live steam being injected into the digester to maintain an elevated temperature and to maintain agitation of the material.

Following the digester treatment, the material is transferred to neutralizing tanks where suitable agitation is maintained by means of a stirrer or injected air. In the neutralizer



## Utilization of Industrial Waste and By-Products

tanks a reagent such as sodium hydroxide is added to neutralize the acid and form a salt which is not harmful to the end product. The pH is raised to the range of 5 to 6 and preferably about 5.5. Sodium chloride formed by the neutralization of HCl with addition of NaOH is desirable in that it acts as a preservative in the end product. The quantity of moisture is subsequently reduced to a range of 15 to 30% and for this concentration the amount of salt inherently produced serves as a preservative. The salt content is preferably about 16 to 20% depending and based upon the moisture content of the final emulsion product.

The material is then transferred to a stabilizer and emulsifier tank to stabilize the emulsion formed between the protein derivatives in aqueous solution and the liquified fats. It will be understood that the protein derivatives are amphoteric in character. The molecules are hydrophilic and lypophilic and therefore able to attract both the water and the fat molecules to emulsify the material. This is fairly stable, but stabilization is improved by the addition of other edible emulsifying agents such as glycerol monostearate. The fat portion of the emulsion is stabilized to prevent rancidity by such agents as butylated hydroxy toluene and/or butylated hydroxy anisol and/or ethyl or propyl gallate and/or lecithin, and other agents. The fat stabilizing agents mentioned are preferably used in combination with each other because the effect of the use of combination of these agents is to form greater fat stabilities than if any of them were used individually.

After neutralization and stabilization, the product is screened by fine screens which normally are 40 to 100 mesh, or it is filtered or impurities are removed by centrifugation or other mechanical means. Thereafter, evaporation is effected. If an open atmospheric pressure evaporator is used, instability of the end product sometimes results in that the fat separates from the proteins because of high temperatures in the range of 240° to 250° F. and temperatures at the steam coil on the order of 350° F. Hence, it is preferable to use a vacuum evaporator or a vacuum pan where the end temperatures are in the order of 135° to 140° F. The vacuum pan concentrates the material to a desired moisture content in the order of 28 to 30% water although this may be reduced to 15 to 20%, depending on the salt content. The resulting product has the viscosity of apple butter or axle grease.

This consistency enables the material to be pumped readily and it can be metered through volumetric meters to determine quantities for batch mixes or even continuous mixes in the feed mills. It is held in a standardization tank where it cools and where it can be analyzed and reblended with other materials in order to meet the standards of the finished products. From this tank the material is pumped to final storage from which shipments are made to mills for incorporation of the product into grain and oil cake ground mixtures in order to produce either high energy or polished animal feeds.

When mixed with grain, the material is more or less extruded by pumps through a pipe in a sausage-like form into the mixer. This is an improvement over the adding of fats to feed in liquid form (as generally practiced heretofore) where the fats have to be heated to fairly high temperatures and then sprayed under pressure onto the feed while the mixer is running, with consequent lumping. However, when fed in the form of paste, as in accordance with this process, the material does not run into the feed but rolls in the mixture with the feed and is gradually absorbed by rubbing on the particles of feed. This method of mixing prevents lumping. Further, the need of expensive heating equipment such as boilers and spray equipment and the expense of operation to incorporate heated fats into the feed is avoided.



Better distribution through the feed is also accomplished, and the fat is not deteriorated by subjection to continued heating. The end product is mixed with ground grain, such as milo, barley, corn and oil cakes such as soy bean oil or cottonseed oil cake meal. A conventional feed is a mixture of ground grain and oil cake, usually blended with minerals and vitamins and usually fish meal, meat and bone scrap meal and is a rather powdery mixture which raises a great deal of dust and is disagreeable to handle. By contrast a polished feed treated with the material of this process requires only that about 1 to 3% of the material be added. This depresses the dust in the mill and reduces handling problems. It has also been found that the dust suppressing effect of the emulsion is considerably greater pound for pound than that of fat bearing materials heretofore employed.

### Dry Free Flowing Food Supplement from Animal By-Products

A process for obtaining a protein rich feed supplement in dry free flowing form from the acid hydrolysis of animal by-products has been developed by W. Kuster (U.S. Patent 3,301,681; January 31, 1967; assigned to Eagle-Ottawa Leather Company).

"Animal by-products" is meant to include tannery fleshings and hide trim, both green and limed, as well as poultry viscera, trimmings, and the like. The selected animal by-products are deposited continuously or in batches in a hydrolysis chamber. At this stage the animal by-products are subjected to hydrolysis with phosphoric acid in aqueous solution at a pH of less than about 2. Hydrolysis of the protein in the animal by-products is promoted by the application of heat such as by injecting steam into the solution. Suitably, the heat is regulated so that the reaction temperature is less than 250°F. In any event, the heat should be adjusted so that the protein is not adversely affected nutritionally.

Hydrolysis of the protein is continued until the protein reaches approximately the polypeptide stage. (The term "polypeptide" is intended to cover the average condition of the protein material. Thus some of the protein may be in the form of a peptone and just soluble in water while some of the protein may be converted to the peptide stage.) It is then removed in aqueous phase from the hydrolysis chamber and further treated. It may contain undesired solid portions such as hair of the animal by-products which may be separated by filtering or screening.

During the hydrolysis treatment the fat portions of the animal by-products, by reason of the heat applied, become fluid and liquified along with the aqueous protein phase. Since the end product desired is low or fat free, the next step is to separate the necessary amount of fat from the hydrolyzed protein. This may be accomplished by any conventional technique for separating fat from an aqueous phase.

Following the separation of the fat from the aqueous phase, the aqueous phase remains at a low pH due to the presence of the phosphoric acid and monocalcium phosphate during the hydrolysis step. Drying the aqueous phase protein is then commenced. The water is reduced by evaporation by heating the solution with or without the aid of a vacuum. Water reduction is continued as far as possible but is ceased before the protein becomes too tacky to permit physical handling for the succeeding stages to be described. Some water reduction usually occurs during hydrolysis due to the heat utilized. In some cases this reduction may in itself be great enough to preclude the necessity of further water reduction such as by vacuum



evaporation. The amount of moisture that should be left with the protein will vary depending upon the composition of the product. In general the water content of the product at this stage will be in the range of about 7 to 40%. Where the ash content is low it may be necessary to cease water reduction when the water content drops to about 20 to 30%. On the other hand, where the ash content is high, it may be possible to reduce the moisture to as low as 5% or less before the product becomes undesirably tacky.

In any event, water reduction is suitably executed so that the water quantity that remains is low enough to be bound as water of hydration upon addition of the calcium compound in the next stage of treatment, leaving a normal "dry" water content of about 5 to 8%, i.e., the product is rendered commercially dry. Because this quantity is variable and depends upon the starting material, the best method of ascertaining the permissible water content is empirical.

Following water reduction to the appropriate stage, a calcium compound selected from calcium oxide and calcium carbonate (preferably calcium carbonate) is added to the protein in an amount sufficient to bring the pH toward neutrality, i.e., about a pH of 5 to 8 and preferably about 6 to 7. While the calcium compound serves to neutralize the product, the calcium ions present form a dicalcium phosphate salt from the monocalcium phosphate salts present in solution in the protein hydrolyzate composition. The dicalcium phosphate salt contains 2 water molecules of hydration. This water is obtained from the remaining moisture in the composition. When the moisture is so combined with the dicalcium phosphate which is practically insoluble in water, it is no longer free and the product is rendered dry and free-flowing. This, of course, requires that the moisture content had been suitably reduced in the preceding step so that the amount of dicalcium phosphate present is sufficient to combine with enough of the remaining moisture to produce the desired dry state. As noted, this requirement is most conveniently determined empirically.

Aside from the chemical combination of the water, moisture is reduced by boiling caused from the heat of formation of the dicalcium phosphate from the monocalcium phosphate. This heat is uniformly distributed throughout the mass and avoids problems caused by heating the outside of the mass such as increased tackiness. Calcium carbonate is a preferred material since the carbonate ions are lost by conversion to  $\text{CO}_2$  gas during the neutralization. Calcium oxide is conveniently used where foaming is a problem.

Calcium carbonate has a unique advantage in the liberation of  $\text{CO}_2$  gas. As the gas is liberated, it breaks the mass uniformly throughout and permits moisture release. This expansion of the gas disintegrates the tacky mass, allows steam to escape at lower temperatures without requiring pressure build-up, and leaves the product in a desirable granular consistency. The steam and  $\text{CO}_2$  gas release in combination with the partial free moisture loss and the conversion of the calcium phosphate from the soluble state to the insoluble state results in a loose friable meal instead of the starting tacky mass. This granular meal, if desired, can now be subjected to additional drying in conventional equipment which could not be used on the tacky mass.

### High Fat Content Feed Product

D.E. Rothschild (U.S. Patent 3,004,852; October 17, 1961) has developed a process to



## Utilization of Industrial Waste and By-Products

take the standard animal by-products of the meat industry and process them into a suitable animal feed with a high-fat content, which is free-flowing and may be pelletized and consequently readily mixed with other foods. In this process an addition agent of a fat absorbent type is added during the pulverizing process. The procedure generally is to take the by-products of the meat industry consisting of meat, bones and fat and shred them to reduce the mass to particles no larger than one cubic inch in size. This raw material is then placed in a cooker, frequently in batches of perhaps 4,000 pounds, and cooked at a temperature of approximately 240°F. for 2 to 4 hours.

The ground and dehydrated mass is discharged from the cooker by gravity or by any simple conveyor, paddle or scoop action and is placed in a drainage pan having a perforated false bottom so that the solid material will lie on this false bottom and the liquid portion fats drain into the area below the false bottom and be pumped away. In order to speed the draining process, the material may be stirred from time to time. Without the exertion of any pressure upon the mass, the fat content should be stabilized at the end of approximately 30 minutes by the cessation of drainage.

The material is then permitted to return to ambient temperature, at which time processing may proceed. Although lower temperatures are desirable, refrigeration is unnecessary, but forced draft may accelerate the cooling. The material is then fed into a hammer mill or other suitable grinder. The material should be addressed to the grinding area and as closely thereto as can reasonably be achieved, hydrated calcium silicate should be fed to the grinding surfaces in an enclosed system by a vibrating screw, but without any premixing of the unground feed and the hydrated calcium silicate. Any material that has passed through the grinder and achieved a particle size of minus 10-mesh should be removed and the remainder of the material recycled through the grinder both as to the excess hydrated calcium silicate and the cooked materials including fat.

When these materials are returned to the grinder for a second pulverization and when they achieve a minus 10-mesh particle size, they should be removed and those not achieving that particle size should be recycled for a third time at which time they will ordinarily pass through the chosen screen. The product so achieved has all of the desirable characteristics. It may be readily pelletized by ordinary procedures, it has a known fat content and may be mixed with other animal feeds to achieve a ready-to-consume animal feed of known nutritional content. Antioxidizing agents may be added immediately before the final grinding with hydrated calcium silicate. The particle size may be even smaller with the use of suitable smaller mesh screens. The process may be started with the filtered cake which remains in filters after tallow filtration has been carried out. Such filter cake lends itself readily for grinding with hydrated calcium silicate to form a highly stable granular material.

### FEED SUPPLEMENTS FROM FISH INDUSTRY

#### Nonhygroscopic Fish Solubles Product on Vermiculite Carrier

Fish solubles is the term applied to the condensed serous portion of fish from which most of the natural fat or oil has been removed. It has never been successfully dried commercially to a solids content substantially higher than 50% by weight due to the fact that its



## Utilization of Industrial Waste and By-Products

proteinaceous constituents possess such a high degree of hygroscopicity as to preclude its effective drying and particularly its subsequent dry storage.

M.E. Parker (U.S. Patent 3,130,054; April 21, 1964; assigned to W.R. Grace & Co.) is able to produce a unique dry, relatively nonhygroscopic product of greatly increased fish solubles content by mixing increased quantities of condensed fish solubles with relatively lesser concentrations of exfoliated vermiculite. Due to the enormous drying surface areas per unit of volume of exfoliated vermiculite, the moisture in the condensed fish solubles is much more readily removed without resort to the relatively high drying temperatures.

Furthermore, the new product is characterized by its extremely high, surprising and unexpected resistance to any subsequent absorption of moisture vapor even when the vermiculite concentration is in proportionately lesser amounts such as 10 to 20% by weight, for example. In practice these increased concentrations of fish solubles on the vermiculite carrier can be dried in one operation if a drum dryer or a steam-jacketed dry rendering kettle is used.

Example: In producing a product of high fish solubles content, one part by weight of a dry nonhygroscopic fish solubles solids product containing one-half part by weight of vermiculite is first made, and this predried product is then mixed with two parts by weight of condensed fish solubles, followed by drying such recycled mixture to result in a final product containing 80 to 85% fish solubles solids.

Normally, exfoliated vermiculite, 95% by volume of which would be retained on a 30 mesh screen with less than 10% by volume retained on an 8 mesh screen, will enable the drying of a nonhygroscopic fish solubles product containing up to 65% fish solubles solids when using a conventional rotary dryer without having to resort to recycling. However, to increase the fish solubles solids to 80 to 85% by weight, it will be necessary to recycle adding sufficient condensed fish solubles to the predried 50 to 65% fish solubles solids product so as to result in an ultimate final product containing 80 to 85% by weight of fish solubles solids.

### Pellets from Fish Meal and Condensed Fish Solubles

The difficulties involved in pelleting fish meal are overcome in the process of G.T. Lanz (U.S. Patent 3,359,115; December 19, 1967; assigned to Ralston Purina Company) by adding condensed fish solubles and an antioxidant.

Example: From a supply bin of fish scrap or meal the particles of fish scrap are fed by a conveyor to a press where the fish meal is pressed. The pressed fish scrap or meal, upon leaving the press may be fed to a dewatering grinder although such a grinder is optional. The fish scrap is then fed into a rotary dryer. As the fish scrap enters the dryer, condensed fish solubles are delivered from a storage tank and sprayed on the fish scrap. An antioxidant, such as ethoxyquin, may also be added at this point from a supply. The amount of condensed fish solubles added to the fish scrap or meal may be about three parts by weight or less of condensed fish solubles for every seven parts by weight of fish scrap or meal. The temperature within the dryer is usually in the range of 500° to 600°F., but may be higher. A substantial amount of water is evaporated from the mixture while it is in the dryer so that the mixture has about 6 to 8% moisture content upon leaving the dryer. Rather than adding the antioxidant to the fish scrap or meal just as it enters the dryer, the oxidant may be added to the



mixture after it is discharged from the dryer. The mixture of fish scrap or meal, condensed fish solubles and antioxidant is then fed into a grinder where it is ground, reduced in size and further mixed. Upon being fed out of the grinder the mixture usually includes one-sixth to one-fourth pound of condensed fish solubles for every one pound of fish scrap or meal, but these proportions may be varied. Considerable moisture was driven off from the condensed fish solubles in dryer.

The mixture of fish scrap or meal, condensed fish solubles and antioxidant is then delivered to a surge bin from which it is fed by a feeder to a conditioner of a pellet mill. Additional condensed fish solubles may be added to the mixture just as it enters the conditioner. In the conditioner the mixture is heated and conditioned for pelletizing. The additional condensed fish solubles added to the mixture as it enters the conditioner increase the lubricity of the product and act as a binder when the pellets are cooled and dried. A rotating mixer in the conditioner thoroughly mixes the condensed fish solubles added to the conditioner with the mixture supplied by the feeder. Upon leaving the conditioner the mixture is formed into pellets by the pellet mill. The pellet mill is conventional and under pressure produces short cylindrical pellets.

Other shapes of pellets, such as rectangular or oval may be produced if desired depending upon the die used in the pellet mill. During the pelleting process in the pellet mill particles are squeezed through dies which compress and heat the pellets. Strands or lengths of compressed particles are automatically cut to pellet length. As the pellets are ejected or discharged from the pellet mill they are in the form of tacky, slightly moist plastic material. The pellets are in a heated condition, such as in the range of 140° to 180°F, when ejected from the pellet mill and are then passed through a cooler where a blast of air cools the pellets to ambient temperature. The moisture content in the pellets is between 10 and 12% upon leaving the cooler. Upon leaving the cooler the pellets may be delivered to a storage unit.

### Fish Cake from Rough Fish

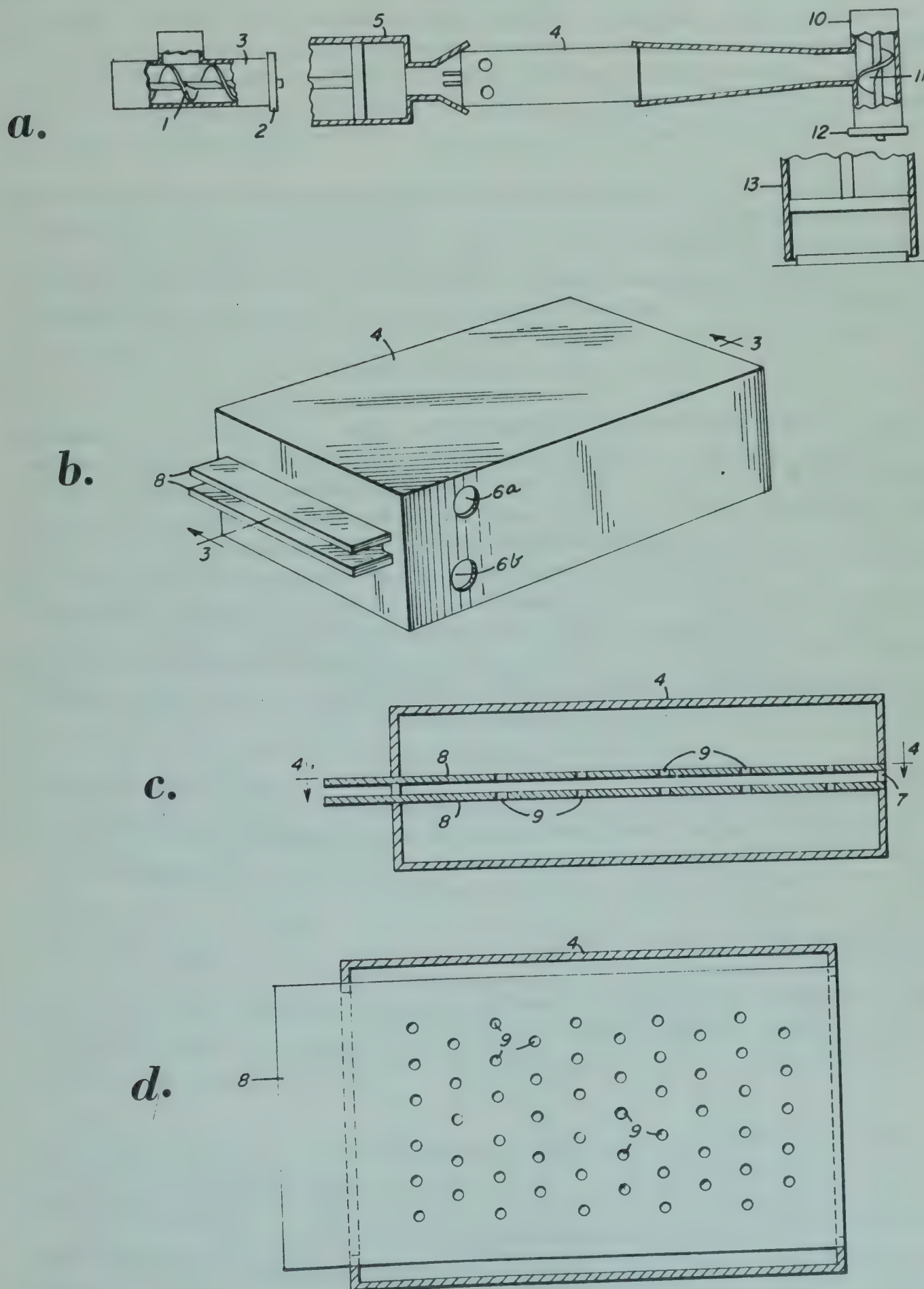
In many regions fish of high human food value have almost disappeared and have been replaced by less desirable species, known as rough fish, not suitable for human food.

R.H. Gnaedinger (U.S. Patent 3,429,710; February 25, 1969; assigned to U.S. Secretary of Interior) provides a method and apparatus for economically processing these rough fish in order to produce a form of fish very desirable to the animal food market.

Figure 14.6a is a schematic diagram showing a system embodying this process. Figure 14.6b shows the cooker. Figure 14.6c shows a side sectional view of the cooker. Figure 14.6d shows a top view of one of the fish-containing plates in the cooker. Referring to the drawings raw fish is conveyed by a helical screw 1 through a perforated plate 2 with 1/4 inch diameter holes in meat-type grinder 3. The ground fish is then fed into the cooking chamber 4 by a feed mechanism such as a piston-type sausage stuffer 5. The rectangular shaped cooking chamber 4 contains heating steam inlets 6a and 6b. Stuffer 5 forces the ground fish between two closely spaced perforated metal plates 8 protruding from chamber 4, the plates spaced apart, for example, 1/2 inch. Fish and steam exit from the chamber through outlet 7. The closely spaced plates extrude the ground fish into a thin layer before it enters the cooking chamber 4, and the plates maintain the fish in a thin layer while it is contacted with jets



FIGURE 14.6: FISH CAKE FROM ROUGH FISH



of live steam delivered uniformly through perforations 9 to both sides of the ground material as it passes through chamber 4. Under this arrangement, the ground material can be heated rapidly to relatively high temperatures to destroy harmful bacteria and enzymes including thiaminase, as opposed to slow heating processes which can result in loss of valuable elements in the fish, and which can increase the formation of undesirable odors and flavors. One pound of raw fish can be heated up to 180°F. in less than one minute during continuous passage through the cooking chamber of this process.

Once the raw fish enters the cooking chamber, the steam pressure within the chamber aids in moving the material through the rest of the chamber past outlet 7. Material emerging from cooking chamber 4 is ground by, for example, a meat type grinder 10 containing a helical screw 11 and perforated plate 12 with 1/2 inch diameter holes. Grinder 10 further makes it possible to regulate the rate of flow of fish through the cooking chamber thereby controlling the rate of cooking. Cooked fish emerging from the grinder 10 falls directly into a press cage 13 and is pressed, for example, for 5 minutes at a pressure of 10 to 15 psig.

To minimize loss of heat during filling and pressing, the cage is insulated. Pressed material in the form of a cake is packed in plastic bags and allowed to cool at room temperature for about 1 hour before being frozen. Immediate packaging of the hot material minimizes contamination of the product during subsequent handling. To enable compression of the fish in the press cage the rough fish must be cooked sufficiently. A minimum temperature of 180°F. for the cooked product is necessary to achieve this. In order to destroy the undesirable thiaminase enzyme that is present in most rough fish, the cooked fish must be held at 180°F. or above for several minutes (e.g., 5 to 10 minutes) during the process. No matter what particular rough fish is employed as the feed material, the press-cake produced by the process is of a relatively uniform composition. Such a nonthiaminase-active, uniform product is more desirable to mink ranchers and other animal food outlets.

Only 1/2 the storage space required by raw fish is required by the press-cake product, and the stored product is more stable than the raw fish since undesirable enzymes, bacteria, etc., have been destroyed by cooking thereby preserving the desirable qualities of the original fish. This capability of the press-cake product makes it highly suitable for storage during glut seasons.

### Stable Fish Meal

The process developed by S. Arakawa, S. Tominaga and T. Terasa (U.S. Patent 3,437,489; April 8, 1969; assigned to Nichiro Gyogyo K.K., Japan) is useful to produce fishmeals of stable and nutritious quality at low cost by preventing the oxidation of fat and oil in fish-meals.

Example: 100 kg. of sardines are ground with a meat grinder having a plate mesh of 6 m./m. and mixed together with 30 kg. of water. The mixture is stirred in a hopper to reduce its viscosity, and is fed to a steam injector by a pump. The fish meat is instantaneously and uniformly heated to 55°C. and falls into a receptacle having a stirrer. Prepared hydrogen peroxide water produced by diluting 120 grams of 30% hydrogen peroxide to be 10 times its volume is gradually added to the fish meat at a constant rate with stirring, and the mixture is pumped into a continuous solid bowl centrifuges to separate liquids from wet solids.



## Utilization of Industrial Waste and By-Products

The wet solids are mixed with a previously dried fishmeal at a ratio of 1:1 to reduce the water content and are dried in a pneumatic conveying dryer to obtain 18 kg. of a fishmeal having a mild smell. Tests showed the fat and oil in this fishmeal to be stable at 30°C. and 70% relative humidity. The taste, odor and color remained stable for a long time. The protein was well digested by fowls.

### Pet Food from Clam Waste

So-called "sea clams" (*Macra solidissima*) have been used for human foodstuffs for some 20 years or more. The portions of the sea clam which are discarded are generally referred to as clam "bellies" and they include the stomachs, livers and other organs. There may be, of course, small amounts of muscles attached which are left in the preparation of the clams as foodstuffs. These parts of the clams, hereinafter referred to as "clam waste," have never before been used.

J. Marvin and E.E. Anderson (U.S. Patent 3,017,273; January 16, 1962; assigned to Pet Kitchens, Inc.) have found that by processing these clam wastes with a thickener, a composition of material may be made which has the desired consistency, texture and taste appeal for a canned animal food. Inasmuch as it is necessary to prevent any bacterial or enzymatic degradation of the clam wastes, it is necessary to process this material soon after its extraction from the meat. It may also be found desirable, although it is not necessary, to grind or otherwise macerate the material before mixing with the thickener.

Example: Clam wastes were heated to a temperature of about 160°F. until the material had been converted into a relatively free-flowing liquid. To this was slowly added a quantity of low methoxy pectin, equivalent to 2% by weight of the clam wastes used. The mixture was maintained at the 160°F. temperature and slowly stirred until the pectin had been thoroughly mixed therein and the composition resulting was a thick liquid of uniform texture and consistency. This thick liquid was then introduced at a temperature of about 160°F. into a size 300 x 407 can (15 to 16 ounces), and the can sealed while the material was maintained at that temperature.

Subsequently the material in the can was sterilized by exposing the can to superheated steam, maintained at 240°F. Sterilization was carried out for about 70 minutes. When the can was subsequently opened the material contained therein was a dark gray, gel-like, finely textured material which could be spooned out or otherwise removed in discrete portions without any crumbling, but also without any adhering of the material to the spoon or to other utensils.

### MISCELLANEOUS

#### Stabilized Fermentation Broths

It has been observed that the fluid fermentation products from fresh whole fermentation broth derived as a by-product from processes such as brewing, cheese making and certain other food refining industries contain substances with growth stimulating factors not due to the known vitamins in their dehydrated and concentrated form. It has been the general practice

## Utilization of Industrial Waste and By-Products

in food processing industries such as brewing, sugar refining, cheese making and others to discard the fluid fraction of fermentation broths or to feed it in fresh form to livestock, kept conveniently close to the plant where it was produced. Fermentable broths are notoriously difficult to store; bacterial and enzymatic action at normal summer temperatures produces rapid and uncontrolled biological reactions which result in making fermentable broths unfit even for animal consumption. Accordingly, most of these fermentation broths are now disposed of as waste or concentrated by dehydration into nonviable products because of the difficulties of storing them in viable forms and shipping them in a viable state for later feeding to livestock.

F.H. Clickner (U.S. Patent 2,965,489; December 20, 1960; assigned to Soluble Nutrients, Inc.) has developed a process of stabilizing the biological activity of viable whole fermentation broth at ambient temperatures by means of adjusting the pH to between 4.00 and 5.80 with the addition of acid and the osmotic pressure to between 85 and 100 atmospheres by the addition of nonpolar water-soluble nontoxic substances. A typical embodiment is described as follows:

From 12 to 40% by volume of whole fermentation broth, and from 55 to 78% by volume of unrefined sugar syrup are added to from 1 to 15% by volume of Vitamin B<sub>2</sub>, B<sub>12</sub> broth. The pH is adjusted from 4.0 to 5.8 (preferably nearly 4.5) with phosphoric or lactic acid.

The osmotic pressure is adjusted by the addition of the sugar fraction to between 85 and 100 atmospheres, preferably near 90 atmospheres. Vitamin additives are used in quantities sufficient to assure preselected concentrations. The osmotic pressure is apparent osmotic pressure computed from dialysis rates through regenerated cellulose membranes of preparations immersed in aqueous solution of glucose at 50°F. for 24 hours.

The whole fermentation broth may be selected from such end products in fermentation processes as wort water derived from beer manufacturing, whey from cheese making, fish solubles and stickwater from fish and meat packing processes. The sugar syrup may be any unrefined sugar containing a solution such as derived from various steps in the processing of corn, beet and cane sugars as well as certain fruit packing and preserving processes. Vitamins B<sub>2</sub> and B<sub>12</sub> are available in economically inexpensive form as whole broth. A specific stabilized broth is:

|                         |       |
|-------------------------|-------|
| Wort water              | 15%   |
| Hydrol                  | 64%   |
| Corn steep liquor       | 6%    |
| Vitamin broth B complex | 15%   |
|                         | <hr/> |
|                         | 100%  |

|                  |         |
|------------------|---------|
| pH               | 4.95    |
| Osmotic pressure | 92 atm. |

Stability: At room temperature no measurable gas evolution after 10 days. At 120° to 125°F. 0.6 ml. gas evolution from an 8 ml. sample after 10 days.



## Utilization of Industrial Waste and By-Products

Stabilized fermentable broths may be stored indefinitely at ambient temperatures with substantially no continued biological activity or deleterious enzymatic reactions, yet all the while remaining in a viable condition. The addition of water and appropriate ambient temperature revive the latent bacteria, molds and fungi whereupon fermentation will again be initiated. The stabilized and enriched fermentation broths are utilized by spraying them onto dried grain fodder prior to feeding, placing them in water in preparation for feeding to livestock, or feeding them directly to livestock in the stabilized form.

In a later process, F.H. Clickner (U.S. Patent 3,272,633; September 13, 1966) utilizes a fermentation broth, not derived as the unstable by-product of industrial wastes as described previously but one which he terms an "active artificial fermentation", prepared from stable commercially available materials. Broadly the preparation of active artificial fermentation broth as a substitute for whole fermentation broth comprises the following steps including the proportional ranges of materials therefor:

An amount of nonlegume cereal product equivalent in starch content to 6 to 8 lbs. of corn starch and 6 to 10 lbs. of barley malt were mixed with 100 gallons of tap water. The non-legume cereal product should preferably be high in starch content.

It will be noted that the amount of barley malt added is equal to the amount required to convert (react) with all starch present in the cereal. The above mentioned three materials are mixed together at ambient temperature to form a fermentable composition or artificial wort water as the first process step.

As the second step of the process the following mixture is prepared: 100 gallons of the fermentable composition from the first step is inoculated with 0.8 to 1.6 pounds of aspergillus oryzae culture (dry basis). The resulting mixture is allowed to ferment at the approximate body temperature of the animal to which the end product food supplement is intended but not exceeding 115°F., until the fermentation rate thereof becomes retarded.

As the third step of the process, a culture of bacillus subtilis, in the amount of 0.4 to 1.2 pounds (dry basis), is added to the fermented mixture obtained from the second step. The resulting mixture is again allowed to ferment at the same temperature as that of the second process until the fermentation rate thereof again becomes retarded.

As the fourth step of the process, 0.6 to 1.0 pounds (on a dry basis) of a lactobacillus culture was added to the fermented mixture obtained from the third step of the process, and the resulting mixture is again allowed to ferment at the same temperature employed in the fermentation during the second and third steps of the process. The aggregate time for the fermentation during the second, third and fourth steps of the process should be about 48 hrs. The resulting composition obtained at the end of the above described fourth step of the process is a viable liquid active artificial fermentation broth and may advantageously be substituted for the previously mentioned industrial by-product. Further processing follows.

To approximately 8,000 gallons of the active artificial fermentation broth is added 200 pounds of riboflavin whole broth (dried culture), 200 pounds of Vitamin B<sub>12</sub> whole broth (dried culture) and enough unrefined sugar syrup (39.5 Brix) to obtain an osmotic pressure of about ninety atmospheres in terms of standard temperature and pressure conditions.



## Utilization of Industrial Waste and By-Products

The acidity in terms of pH value of the previous mixture was usually found to be within the required range of 4.00 to 5.80. Where the pH value is found to be outside the required limits choline chloride may conveniently be used to bring the mixture within the required pH range.

The resulting product is stable for indefinite periods of time at ambient temperatures and is ready for shipment to the ultimate customer (i.e., feed dealers, farmers, etc.). The product thus obtained being stable may be stored with substantially no continued biological activity or deleterious enzymatic reactions, yet all the while remaining in a viable condition. The stabilized and enriched product is utilized by spraying it onto dried grain fodder prior to feeding, or placing the product in drinking water in preparation for feeding livestock or poultry.

### Salt Blocks from Potash Refining Wastes

In various mines and refineries near Carlsbad, New Mexico, a waste by-product of the potash refining process consists of salt containing small amounts of other minerals which are highly beneficial to cattle and stock. The chemical composition of the waste by-product from mines in the above mentioned region is approximately 96% sodium chloride, 3% potassium chloride, 0.5% calcium sulphate, 0.4% magnesium silicate and 0.1% iron oxide, the latter giving the product a distinctive pinkish color. Blocks heretofore made therefrom have absorbed moisture readily, become soft and crumbled easily when exposed to the weather.

The process of L.S. Williams (U.S. Patent 3,066,024; November 27, 1962) provides a method by which the potash waste products above mentioned may be economically and advantageously formed into dense, strong, water-resistant stock salt blocks.

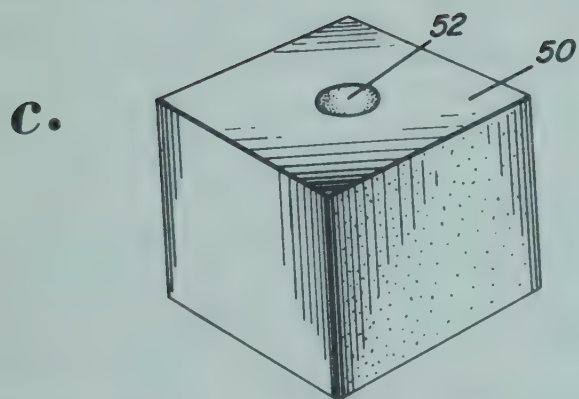
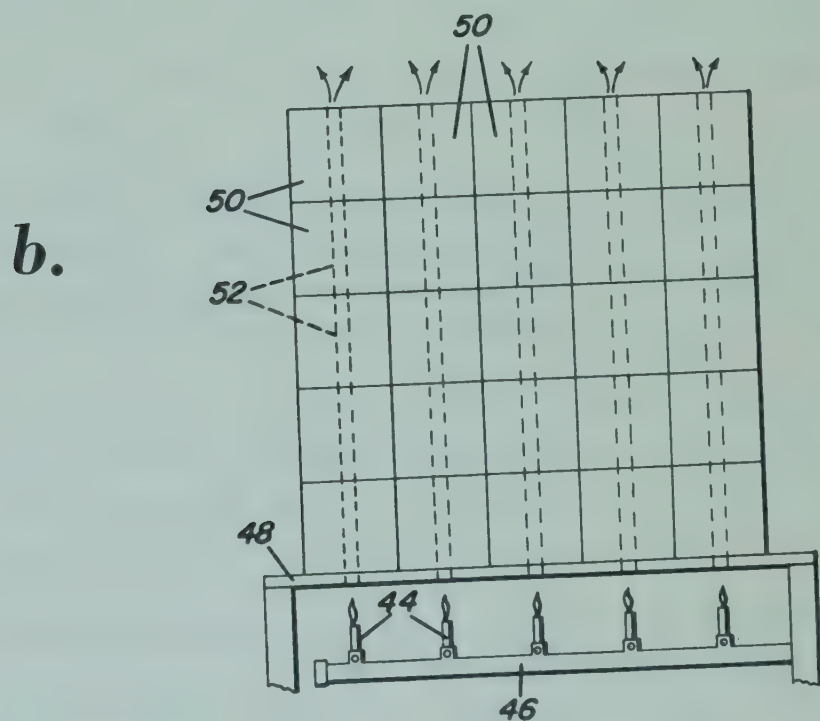
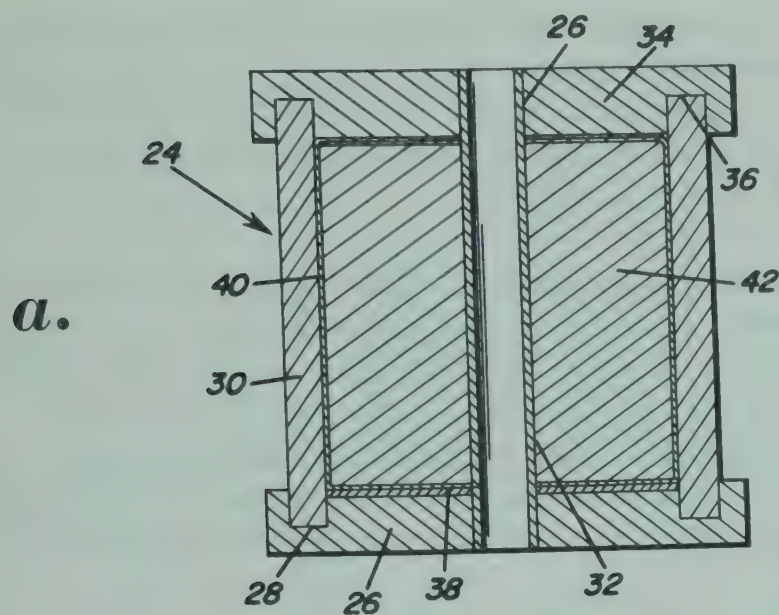
In carrying out the process of forming salt blocks, it is understood that the potash salt obtained as a by-product during the refining of potash from mines or refineries is suitably crushed, and has been delivered as by a discharge means upon a screen, through which the crushed potash salt passes. This screen is preferably 28 mesh, having 28 openings per linear inch or 784 per square inch. After passage through this screen, the particles are collected and deposited in a moistening vat into which a saturated brine solution is discharged; the brine being added until the screened potash salt has a moisture content of from 1 1/2% to about 2% by weight. Suitable means may be provided for stirring the salt in the brine solution to effect thorough moistening of the same.

The brine-moistened, crushed and screened salt is then applied into suitable molds which form a salt block of the requisite shape. A satisfactory form of mold is shown in Figure 14.7a, the mold being indicated generally by the numeral 24. The mold includes a base 26 whose top surface has grooves or channels 28 in which are received the lower ends of the side walls 30. A mold core in the form of a rod or pipe 32 is secured in the base 26 and preferably extends upwardly from about the central portion of the base.

The mold is completed by a top 34 likewise having channels 36 in the lower side thereof which receive the upper ends of the side walls 30. The top wall also has an opening or openings 36 therethrough for sliding engagement upon the upper end of the upstanding mold core 32.



FIGURE 14.7: SALT BLOCKS FROM POTASH REFINING WASTES



Within the mold cavity, and resting upon the bottom wall 26 of the mold there is provided a sheet of asbestos 38 of the same size as the interior of the mold and likewise provided with an opening to receive the mold core 32 therethrough. The inside of the mold cavity is lined with a protective sheet such as heavy kraft paper, this lining being indicated by the numeral 40 and extending along each of the side walls, upon the bottom wall 26 and the asbestos sheet 38 thereon, and being of sufficient height to be folded in upon the body of moistened salt 42 with which the mold cavity is then filled.

The body of moistened salt is tamped or otherwise packed in the mold in any suitable manner and under moderate pressure as for example by hand tamping. This operation serves to compress and densify the moistened salt, tending to destroy any capillaries which might be formed between the salt particles. The cover 34 is then applied, whereupon the final stage of tamping or compressing of the salt may be effected. Thereafter, the cover is removed, and the body of salt 42 still retained within its protective sheet 40 of kraft paper and still seated on the asbestos pallet 38 beneath the same is removed from the mold 24 as by being slid off the mold core 32.

As now shown in Figure 14.7b, the blocks of compressed salt, still encased in the kraft paper and seated on the asbestos sheet are placed in a kiln of any desired character, the same having a plurality of nozzles 44 from which gases of combustion are discharged, these nozzles being serviced by a suitable supply pipe 46. These nozzles are disposed beneath the supporting platform 48 upon which the salt blocks each designated by the numeral 50, see also Figure 14.7c, are stacked in any suitable relation. It will be observed in the stacking the central aperture or openings 52 through the block left by the mold core 32 are disposed in alignment as shown in Figure 14.7b to provide when the blocks are in juxtaposition continuous passages through the stack of blocks when piled upon the platform 48.

It will be observed that the stacks of aligned blocks are so disposed that the aligned passages 52 will be each disposed over one of the burners 44 from which the products of combustion pass upwardly through corresponding openings in the platform 48 and through the continuous passages to be discharged at the top thereof as shown by the arrows.

In this manner, the heating medium is applied directly to the center of each block and heats the block from the center outwardly, thereby expelling or driving the moisture before it from the center of the block to the outer surfaces thereof. In this manner the prior art difficulty of leaving a block with a relative wet or spongy center, effected by heating the blocks from the exterior thereof, is completely overcome so that the block is of uniform dryness throughout, and thus attains its maximum strength.

The heat which is applied only to the interior of the blocks through the holes in the center thereof maintains a temperature of about 400°F. at the top of the holes of a stack of blocks which are five blocks high. This heat is applied until the outside surface of the block reaches a temperature of about 340°F. inside the paper wrappings. After the heating operation is completed, and all moisture has been driven from the blocks, the blocks are removed from the kiln and are stored. They require no curing and are ready for immediate shipment, storage and use. The kraft paper is left on the blocks until they are placed in the pasture for the cattle. This paper is made of wood pulp and will not injure cattle, even if they should eat it. However, the paper is easily removed if desired.



### Spent Sulfite Wood Liquor as Binder for Pelleted Feeds

A pelleting process for animal feeds utilizing, as a binder, waste liquors derived from the digestion of plant products, such as wood; citrus pulp; straw from flax, cereal grains and the like; bagasse (cane pulp); sorghum; corn; etc.; as best exemplified by alkaline earth base spent sulfite wood liquors has been developed by C.B. Knodt (U.S. Patent 3,035,920; May 22, 1962; assigned to Cargill, Inc.). A typical calcium base spent sulfite wood liquor derived from the sulfite process in the production of pulp from woods essentially of the hardwood species is commercially available from Marathon Division of American Can Company in dried form under the trademark "Norlig-A" and in liquid form under the trademark "Norlig-L".

The feed ingredients are mixed in conventional feed mixers. The various feed ingredients, along with the sulfite liquor binding material, in either liquid or dried form, are intimately and uniformly intermingled and admixed. The feed ingredients, in most instances, are finely divided and in the form of a dry powdery mash. When admixed in dry form, the calcium base spent sulfite wood liquor is in the form of a fine dry powder. The binder is added to the feed mixture in amounts ranging from about 1/4% to about 5% of the total feed weight on a dry weight (5% moisture) basis. When the binder is incorporated in liquid form, approximately twice as much binder is employed to equal the equivalent binding power of dried sulfite liquor.

The admixed feed and binder are subjected to steam just prior to pelleting. This adds approximately 1 to 10% moisture to the mixture to raise the total moisture content to from 10 to 20% and raises the temperature to between 50° and 150° F. The feed mix is then subjected to pelleting. The feed mix is applied to the top surface of the die plate and forced under pressure of rotating rollers into and through the die openings, being compressed and compacted in the course of its passage and then cut off into segments of desired length by a knife moving against the bottom surface of the die plate. Alternatively, pelleting may be accomplished by other extruding machines, balling drums, granulators, and the like.

After pelleting the feed is cooled, usually by blowing air through the pellets as they pass on a foraminous conveyor. In cooling the pellets lose moisture and become hard and dry. The use of the calcium base spent sulfite wood liquor binder produces pellets having good "green" strength which withstand handling during processing without breaking, deforming, sticking and agglomeration of a plurality of pellets and with minimum production of fines, dusts, etc. The calcium base spent sulfite wood liquors in both liquid and dried form are useful as binding materials in pelleting a great variety of feed ingredients normally and conventionally used in the feeding of livestock, pets and other animals.

Example: A pelleted cattle diet formula has been prepared as follows: A mixture is made up of 40 lbs. of dried calcium base spent sulfite wood liquor, 1,860 lbs. of ground yellow corn, 20 lbs. of dicalcium phosphate, 10 lbs. of ground limestone, 20 lbs. of iodized salt, 50 lbs. of 44% protein soybean oil meal. These ingredients are admixed, moistened with steam and forced through a pelleting die, cut into lengths and blown with air while carried on a screen to harden and dry the pellets.



### Starch Hydrolysate Liquor-Vegetable Meal Dry Feedstuff

Starch hydrolysate liquors, e.g., mother liquors from the manufacture of crystalline dextrose, are particularly useful as soluble carbohydrates in feedstuffs, but heretofore no satisfactory method of mixing such carbohydrates with gluten meal in the desired amount has been found.

M.L.E. van Tittelboom (U.S. Patent 3,372,032; March 5, 1968; assigned to Corn Products Company) discovered that about 50 to 60%, dry basis, of starch hydrolysate liquors, may be mixed with vegetable protein meal to form a dry, powdered product of high carbohydrate content. The preferred ratio of vegetable protein meal to starch hydrolysate liquor is 45 to 50% (meal) to about 55 to 50% (liquor). After all the liquor has been added to the gluten meal the resultant mixture, which is a sticky mass, is agitated slowly until crystallization of the dextrose has been completed. Inasmuch as heat is generated by crystallization of the dextrose, cooling means should be provided to keep the product at a temperature favorable to crystallization.

Example: 4,500 kg. of corn gluten meal having a moisture content of 9% was placed in a vertical conveyor mixer (10,000 liters volume) operating at 60 rpm. A pipe with a spray nozzle was attached to the bottom part of the mobile arm (operating at 1.33 rpm) in order to introduce the starch hydrolysate liquor under pressure and spray the same onto the gluten meal. Five thousand kilograms of mother liquor from the manufacture of crystallization of dextrose which had been reconverted to a DE value of 83 to 84% and having a density of 43.5 Bé. (83% dry substance) was sprayed onto the gluten meal while the same was slowly agitated. The temperature of the liquor was 80°C.

The spraying was done in two stages, although this is not necessary. In the first stage 3,250 kg. of the liquor was sprayed onto the gluten meal while it was agitated during a period of 1.5 hours. Thereafter, the resultant mixture was agitated for 0.5 hour. Then 1,250 kg. of liquor was sprayed onto the mixture during a period of 1 hour. At the end of this period, the mixture was a sticky mass. Agitation of the mixture was continued for 19 hours, the temperature of the mixture being maintained at 38° to 40°C., cool air being introduced as required at the top of the mixer so that the top layer of the mixture was exposed to the cool air. At the end of this period, the sticky mass had changed into a dry powdery mass which was cooled, screened, and packed. The moisture content of the product was about 13%, hence no drying was required. The tailings from the screening operation can be ground and recycled, e.g., mixed with the crystallized product at the outlet of the mixer.

The above method was repeated but all of the starch hydrolysate liquor was added in one stage, over a period of 3.5 hours. Equally satisfactory results were obtained. When the last mentioned method is repeated using a liquor having a DE value of about 78% substantially the same results are obtained. Similarly when deoiled soya bean meal and deoiled peanut meal containing not more than 10% oil are used with either of the aforementioned starch hydrolysate liquors, similar results are obtained.

### Use of Bleaching Earth from Decolorizing of Fatty Oils

H.E. Prückner and R.F.M. Schanze (U.S. Patent 3,340,065; September 5, 1967; assigned to



## Utilization of Industrial Waste and By-Products

Flachsroste Berching GmbH, Germany) have a process for an animal foodstuffs formed by utilizing bleaching earth containing raw fats obtained as industrial waste in the decoloring of fatty oils as a major component and mixing this with cellulosic fibers, e.g., save-all waste fibers or waste paper, to form the free-flowing fodders of high fat contents.

The bleaching earth, for example a mixture of Al and Mg silicates, is used in large quantities for decoloring dark, fatty oils and thereby becomes enriched to a comparatively great extent so that it leaves the filter press or similar plants with a content of raw fat amounting to about 40 to 50%. As the fats occurring are generally highly digestible but on the other hand the bleaching earth component itself is tasteless and exerts an antilaxative effect and furthers the intestinal flora, this fodder is very valuable for feeding animals.

Crude fibers from the manufacture of cellulose and paper are used as cellulose carrier substances. The very fine fibrous stuff from the save-all occurring in the manufacture of high grade cellulose and paper products which is composed of very fine separate fibers and is therefore characterized by its exceptionally large surface area, is particularly useful (as are paper and cardboard). Crude fibers are, in the case of ruminants and also in the case of pigs, converted by a bacterial process and rendered energetically useful.

The carrier substance is finely ground and if necessary freed from incrustations. The bleaching earth is dried and finely ground so that it is as far as possible free from water. In this state it is easy to mix with the cellulose carrier substance and a binding of these two components produces a free-flowing mixture with a content of crude fat amounting to about 45%.

Example: The excellent suitability of the mixed foodstuff of this process for the production of any desired nutritive combination is shown in the following table.

|                                  | Fat,<br>percent | Protein,<br>percent | Starch<br>units |
|----------------------------------|-----------------|---------------------|-----------------|
| 20% Bleaching earth.....         | 10              | -----               | 240             |
| 20% Molasses.....                | -----           | 9                   | 95              |
| 20% Soybean.....                 | -----           | -----               | 140             |
| 30% Stuff from the save-all..... | -----           | 26                  | 165             |
| 10% Urea.....                    | -----           | -----               | -----           |
|                                  | 10              | 35                  | 640             |

### Livestock Feed from Citrus Waste

E. George (U.S. Patent 3,118,768; January 21, 1964; assigned to A.O. Smith Harvestore Products, Inc.) has developed a method of treating organic material, such as acidic fruit and other vegetable waste materials, which are often by-products of commercial canneries, to produce a suitable long lasting feed for livestock.

The waste which results from the extraction of juices in the canning operation generally contains varying quantities of peel, rag, seed, and moisture. This citrus waste material ordinarily can be expected to contain from 80 to 90% moisture and to have a bulk density of about 30 to 33 lbs. per cubic foot. The pH of citrus waste ordinarily is initially in the range of about 4 to 5. The bulky citrus waste is loaded into a modified hammer mill or food chopper where it is ground, shredded, or comminuted to a size of about 3/8 of an inch, although



particle sizes of up to an inch are suitable. The citrus waste as received from a cannery is generally a nonhomogeneous mass and is broken down to this size range primarily to facilitate the economic drying of the mass by establishing a relatively uniform particle size which will expose the largest possible surface area to the drying atmosphere while reducing to a minimum or eliminating the undesirable formation of fines.

When the desired size range has been obtained, the comminuted material is withdrawn from the mill or chopper and is then placed in a drying apparatus. This apparatus is preferably a rotary type dryer in which the shredded material may be continuously tumbled and subjected to a heated atmosphere within the drying chamber. The temperature of the atmosphere within the dryer should be preferably maintained in excess of  $100^{\circ}\text{C}$ . until the moisture level desired has been attained. Care must be taken during the drying operation in the application of heat to the shredded mass being dried to prevent charring and scorching of quantities of the material as this may deleteriously affect the food value and palatability of the feed derived from the citrus waste.

It has been found that the application of heat to the shredded or chopped waste is instrumental in driving off deleterious volatile substances which are believed to be terpene hydrocarbons, such as d-limonene. These substances under certain feeding conditions impart an undesirable flavor to milk when fed to dairy animals and are generally thought to make citrus waste, and in particular orange waste, less desirable as an animal feed.

The drying or evaporative process is continued until the pulp mass has reached a moisture content in the intermediate range of 30 to 70% where previously it was believed necessary to reduce the moisture level to approximately 10% for storage of the treated waste without spoilage. These moderate moisture levels of 30 to 70% can be rather rapidly and economically reached with conventional drying equipment. In reducing drying time and heat input, there is, in addition to a realization of a substantial cost saving, a considerable reduction of the exposure time of the mass in the dryer to heat which thereby greatly reduces the possibility of charring or scorching.

When the desired moisture level has been attained (and this level is usually determined by factors such as shipping distances, available storage area, and diet requirements), the processed waste is removed from the dryer and as quickly as possible, preferably in not more than 24 hours, is loaded into a storage structure or container which is sealed from the atmosphere or adapted to prevent the entrance of oxygen. The structure should be capable of being maintained during the storage period in a substantially oxygen-free condition.

Shortly after the processed waste has been introduced into the storage structure or container, a rapid enzymatic reaction takes place which converts substantially all of the oxygen initially present in the storage area to  $\text{CO}_2$ . For best results, the storage structure should be sufficiently sealed to prevent additional oxygen from entering the structure following the enzymatic reaction. No more than 2% of free oxygen should be permitted within the structure at any time after the enzymatic reaction has taken place. Shortly after the  $\text{CO}_2$  has been produced within the structure, all further microorganic activity is halted or substantially retarded. It is believed that the production of  $\text{CO}_2$  acts to bring the atmosphere within the structure or container to an equilibrium point which inhibits any further activity within the structure. Therefore, the food value and palatability of the stored material may be



## Utilization of Industrial Waste and By-Products

maintained in essentially the same condition it was in prior to the time of drying or evaporation. By maintaining processed vegetable waste in an essentially sealed storage structure or container, the waste may be processed at a tremendous saving in labor, time and expense as the material need only be reduced in moisture content to an intermediate moisture range. By confining the vegetable material in oxygen-free storage, waste or spoilage of the stored feed material in storage may be virtually eliminated.

## COMPANY INDEX

- Abbott Laboratories - 179  
 Activator, Inc. - 61  
 Advance Growth Capital Corp. - 38  
 Agway, Inc. - 202  
 Ajinomoto Co., Inc. - 301  
 Allied Chemical Corp. - 4, 265  
 Allied Mills, Inc. - 254  
 American Cyanamid Co. - 116, 117, 118  
     119, 120, 121, 122, 126, 127, 228,  
     230, 246  
 Archer-Daniel-Midland Co. - 263  
 Armour and Company - 204  
 Beacon Milling Company, Inc. - 216  
 Blackstrap Dry, Inc. - 65  
 Bureau d'Etudes Armand Malchair SA -  
     21  
 Cal-Tam Research Products Corporation -  
     323  
 Canadian Patents & Development Ltd. -  
     274  
 Cargill, Inc. - 55, 339  
 Celanese Corp. - 187  
 Central Soya Co., Inc. - 50, 53, 55, 68,  
     71, 225, 288  
 Ciba Corporation - 205  
 Commercial Solvents Corp. - 59, 63, 89,  
     113, 139, 236, 237, 273, 274  
 Corn Products Co. - 34, 35, 226, 284,  
     340  
 Darling & Co. - 157  
 Dawes Laboratories, Inc. - 110, 142, 212  
 Dawson Chemical Co., Inc. - 148  
 Denver Chemical Mfg. Co. - 99  
 Diamond Shamrock Corp. - 162  
 Du Pont - 171, 189, 191, 261  
 Eagle-Ottawa Leather Co. - 314, 326  
 Eastman Kodak Co. - 56  
 Erly-Fat Livestock Feed Co. - 33, 152  
 Farbenfabriken Bayer AG - 165  
 Farbwerke Hoechst AG - 131  
 Feed Service Corp. - 36  
 Flachsroste Besching GmbH - 341  
 Flavor Corporation of America - 5  
 FMC Corporation - 29, 254  
 Fuu AG - 181  
 Gebruder Giulini GmbH - 149  
 Geigy Chemical Corp. - 175  
 General Foods Corp. - 280, 287  
 Grace, W.R. & Co. - 68, 329  
 Grain Processing Corp. - 106, 107, 221  
 Heterochemical Corporation - 231, 234  
 Hoffmann-La Roche Inc. - 176, 223, 275  
 Hoffman-Taff, Inc. - 224  
 International Minerals & Chemical Corp. -  
     150, 262  
 International Stock Food Corp. - 3  
 Iowa State Research Foundation - 100, 250,  
     251, 278  
 Kaken Kagaku K.K. - 7  
 Katakura Industry Co., Ltd. - 301  
 Kentucky Research Foundation - 271  
 Kosch Co. - 30  
 Laboratories for Applied Biology Ltd. - 292  
 Lancaster Laboratories, Inc. - 24  
 Lilly, Eli & Co. - 97, 100, 172, 217, 247  
 Limestone Products Corporation of America -  
     259  
 Loomix, Inc. - 71  
 Mattox & Moore, Inc. - 103  
 Michigan State University - 27  
 Monsanto Co. - 76, 143, 144, 145, 146,  
     169, 206, 207, 208, 219, 238, 239



## Company Index

- Moorman Mfg. Co. - 35, 166, 270  
 Morrell, John & Co. - 7  
 Nagase & Co., Ltd. - 114  
 National Dairy Products Corp. - 160  
 National Distillers & Chemical Corp. - 74  
 Nebraska Consolidated Mills Co. - 268  
 Netex Mink Ranches, Inc. - 307  
 Nichiro Gyogyo K.K. - 332  
 Nipak, Inc. - 264  
 Nopco Chemical Co. - 195, 209  
 Norden Laboratories, Inc. - 240  
 North American Philips Co., Inc. - 222  
 Northern Trust Co. - 84  
 Norwich Pharmacal Co. - 190  
 Olin Mathieson Chemical Corp. - 95, 204  
 Organic Nutrients, Inc. - 200  
 Pabst Brewing Co. - 108, 131, 258  
 Penick S.B. & Co. - 130  
 Pet Kitchens, Inc. - 333  
 Pfister Chemical Works, Inc. - 316  
 Pfizer, Chas. & Co., Inc. - 86, 101, 103,  
     109, 124, 133, 134, 168, 178, 182,  
     210  
 Pillsbury Co. - 154  
 Polychemical Laboratories, Inc. - 179  
 Pronit Internacional SA - 243  
 Publications, T.F.H., Inc. - 298  
 Purdue Research Foundation - 9, 87  
 Quaker Oats Co. - 281  
 Ralston Purina Co. - 64, 183, 303, 329  
 Recherche et Industrie Therapeutiques - 128  
 Refining Inc. - 48, 51  
 Rosner-Hixson Laboratories, Inc. - 98  
 Saint-Gobain, Compagnie de - 147  
 Separator, Aktiebolaget - 318  
 Sharples Corp. - 43  
 Shell Oil Co. - 137, 138  
 Simmons Milk Products, Inc. - 152  
 Smith, A.O. Harvestore Products, Inc. - 341  
 Smith Kline & French Laboratories - 180  
 Società Farmaceutici Italia - 140  
 Societe des Usines Chimiques Rhone-Poulenc -  
     129, 170  
 Soluble Nutrients, Inc. - 334  
 Sperry Rand Corp. - 13  
 Standard Alfalfa Milling Co. - 18  
 Standard Brands, Inc. - 77, 79  
 Sterling Drug Inc. - 184  
 Summer Iron Works, Inc. - 26  
 Swift & Co. - 52, 115, 290  
 Takeda Chemical Industries, Ltd. - 300  
 Toyama Kagaku Kogyo K.K. - 173  
 Trans-Oceanic - 19  
 Union Carbide Corp. - 266  
 U.S. Atomic Energy Commission - 213  
 U.S. Secretary of Agriculture - 40, 50, 94,  
     95, 111  
 U.S. Secretary of Interior - 330  
 Universal Oil Products Co. - 16  
 Upjohn Co. - 101, 135, 136  
 Vitamins, Inc. - 60  
 Vy Lactos Laboratories, Inc. - 66, 67  
 Weaver, Victor F., Inc. - 317  
 Wenger Manufacturing, Inc. - 39  
 Whitmoyer Laboratories Inc. - 185  
 Wilson and Company, Inc. - 242, 283  
 Wisconsin Malting Co. - 2, 193, 194

## INVENTOR INDEX

- |                          |                                    |
|--------------------------|------------------------------------|
| Abbey, A. - 119          | Burgess, H.M. - 287                |
| Aeschlimann, J.A. - 275  | Burroughs, W. - 100, 250, 251, 278 |
| Akers, W.T. - 246        | Cairney, T.J. - 108                |
| Algeo, J.W. - 71         | Caldwell, M.J. - 35, 166           |
| Anderson, E.E. - 333     | Card, P.Q. - 84                    |
| Anderson, P.C. - 36      | Cardon, B.P. - 152                 |
| Andrews, F.N. - 9, 87    | Castle, C.P. - 15                  |
| Andriuli, F. - 195       | Chaney, C.H. - 271                 |
| Anthony, W.B. - 319      | Chang, T.S. - 185                  |
| Appleman, M.D. - 73      | Chenicek, J.A. - 16                |
| Arakawa, S. - 332        | Ciegler, A. - 40                   |
| Arnold, A. - 184         | Clayton, B. - 47, 48               |
| Axelrod, H.R. - 298      | Clickner, F.H. - 334, 335          |
| Ayres, A.U. - 43         | Clovis, M.A. - 60                  |
| Bachman, M.C. - 236, 273 | Coombes, A.I. - 283                |
| Baker, J.H. - 19         | Couch, J.R. - 197                  |
| Barchielli, R. - 140     | Craig, L.E. - 264                  |
| Barnhart, C.E. - 271     | Craven, W.W. - 225                 |
| Bauer, F. - 131          | Crosby, D.G. - 266                 |
| Bauernfeind, J.C. - 176  | Cunningham, H.M. - 274             |
| Belasco, I.J. - 261      | Czarnetzky, E.J. - 3, 283          |
| Bender, W.L. - 97        | Dawe, V. - 212                     |
| Bergy, M.E. - 135, 136   | Dawson, R.B. - 34                  |
| Berruti, R. - 231        | De Boer, C. - 135                  |
| Bickoff, E.M. - 94, 95   | De Luca, A.P. - 195                |
| Biehl, H. - 10, 11       | Dennis, E.W. - 184                 |
| Bogdonoff, P.D. - 237    | Dereniuk, S.N. - 142               |
| Bone, D.P. - 281         | De Renzo, E.C. - 127               |
| Booth, A.N. - 94, 95     | De Zeeuw, J.R. - 86, 103, 168      |
| Bosin, W.A. - 154        | Docken, I.M. - 308                 |
| Bousquet, E.W. - 171     | Donovan, G.A. - 86, 133            |
| Branom, J. - 18          | Doody, F.X. - 157                  |
| Brent, B.J. - 99         | Edwards, R.B. - 204                |
| Briggs, W.C. - 26        | Eggert, R.G. - 246                 |
| Brooks, J.W. - 258       | Eichenberger, K. - 205             |
| Brown, W.L. - 7          | Elbreder, C.H. - 291               |



# Inventor Index

- Elenbogen, G.D. - 60
- Eller, J.C. - 148
- Elliott, R.F. - 122
- Ellison, T. - 180
- Elmslie, W.P. - 35
- Ely, C.M. - 162, 195, 209
- Ensley, C.O. - 241
- Erwin, E.S. - 238, 239
- Eshleman, J.C. - 199
- Everett, J.P., Jr. - 183
- Eyssen, H. - 128
- Faustini, R. - 140
- Fayhee, P.E. - 12
- Fetzer, W.R. - 77, 79
- Filstrup, P. - 318
- Flechsigg, A. - 19
- Flier, R.J. - 303
- Forest, J.G. - 3
- Forgacs, J. - 228, 230
- Fortenbaugh, R.B. - 119
- Foster, R.O. - 98
- Freese, T.E. - 4, 265
- Friedman, I.J. - 109
- Fujiwara, T. - 173
- Galler, W. - 232, 233
- Gard, D.I. - 217
- Garibaldi, J.A. - 111
- Garner, G.B. - 240
- Garre, B. - 149
- Gasparini, G. - 140
- Gassner, F.X. - 95
- Geerlings, P.J. - 270, 277
- Gehrt, A.J. - 35
- Gelsendorf, A. - 223
- George, E. - 341
- Gershon, H. - 316
- Gerzanich, M. - 292
- Gillis, M.G. - 150
- Gnaedinger, R.H. - 330
- Goldblatt, L.S. - 50
- Golub, S.J. - 60
- Gordon, R.S. - 169, 206
- Gray, R.E. - 52
- Greenberg, R.A. - 115
- Groschke, A.C. - 202
- Guidarelli, E.J. - 55
- Haas, W.J. - 101, 134
- Haase, J.A. - 17
- Hale, D. - 303
- Hallinan, F.J. - 283
- Hamamura, Y. - 300
- Hansen, L.I. - 263
- Hanson, A.M. - 107
- Harrel, C.G. - 154
- Harris, R.C. - 34
- Harrop, L.D. - 101
- Harvey, M.J. - 126
- Hashimoto, S. - 7
- Hayashiya, K. - 300
- Hays, V.W. - 278
- Henson, J.N. - 237
- Hereld, P.C. - 179
- Herman, L.G. - 242
- Herr, R.R. - 136
- Hess, E.H. - 13, 24, 317
- Hickey, F.D. - 254
- Hidy, P.H. - 89
- Hinton, A.V. - 292
- Hochberg, M. - 209
- Hochstein, F.A. - 182
- Hodge, E.B. - 89
- Hoffman, H.E. - 171
- Hoffman, W.H. - 224
- Hollenbeck, C.M. - 2, 193, 194
- Hoover, F.W., Jr. - 26
- Hopper, J.H. - 204
- Huber, G. - 131
- Huhtanen, C.N. - 121
- Hutchinson, H.D. - 270
- Iacono, P. - 160
- Ide, S. - 301
- Ijichi, K. - 111
- Inagaki, T. - 173
- Jarowski, C.I. - 210
- Jaworski, E.G. - 144, 145
- Jensen, C.C. - 157
- Johnson, H.E. - 266
- Johnson, W.P. - 122
- Julou, L. - 170
- Kaemmerer, K. - 165
- Kamlet, J. - 187, 191
- Kato, K. - 301
- Kelley, J.A. - 68
- Kichline, T.P. - 208
- King, W.N. - 29
- Kirk, L.D. - 40

# Inventor Index

- Klein, H.C. - 209  
 Kline, G.B. - 247  
 Klothen, I. - 116, 117, 119  
 Knodt, C.B. - 339  
 Kodama, Y. - 173  
 Koonz, C.H. - 115  
 Kosch, M.A. - 30  
 Kruse, N.F. - 53, 55, 68, 71, 225  
 Kuster, W. - 314, 323, 326  
 Kviesitis, B. - 66  
 Lambert, G.F. - 179  
 Landy, J.J. - 301  
 Lanz, G.T. - 64, 329  
 Lee, C.E. - 216  
 Legator, M. - 137, 138  
 Lent, A. - 33  
 Lewis, J.C. - 111  
 Lewis, R.W. - 201  
 Lillehoj, E.B. - 40  
 Lindburg, R.K. - 211  
 Linskey, E.F. - 284  
 Livingston, A.L. - 95  
 Lobel, M.J. - 167  
 Locuratolo, P.S.J. - 147  
 Loomis, R.I. - 71  
 Ludington, V.D. - 280  
 Ludwig, N.H. - 97  
 Luther, H.G. - 124, 133, 168  
 Lyons, J.W. - 76  
 Machlin, L.J. - 169, 219  
 MacMillan, M.J. - 162  
 Malchair, A. - 21  
 Malzahn, R.C. - 107, 221  
 Marco, G.J. - 143, 238, 239  
 Margot, A. - 175  
 Martin, E.G. - 109, 134  
 Martin, J.L. - 236, 237, 273, 274  
 Marvin, J. - 333  
 Mattox, W.E. - 103  
 McCutchan, W.N. - 113  
 McKeen, J.E. - 101  
 McLellan, W.N. - 95  
 Means, T.M. - 100  
 Mecho, J.P. - 243  
 Mellentin, R.W. - 287  
 Metivier, J. - 170  
 Miller, F.D. - 74  
 Miller, J.P. - 179  
 Minami, Z. - 114  
 Mohile, R.E. - 280  
 Molitorisz, J. - 27  
 Monroe, C.H. - 110  
 Morehouse, A.L. - 221  
 Motzel, W.J. - 245  
 Moyle, R.A. - 310  
 Moyle, R.H. - 310  
 Muller, S.A. - 118  
 Mustakas, G.C. - 40  
 Naito, K. - 300  
 Nakai, M. - 114  
 Nakano, N. - 215  
 Nanninga, J.B. - 234  
 Okada, K. - 301  
 Olson, W.A. - 178  
 Ootaka, K. - 114  
 Pack, F.C. - 50  
 Parker, M.E. - 329  
 Patterson, E.B. - 52  
 Paul, M.F. - 190  
 Peeler, H.T. - 262  
 Pensack, J.M. - 236, 273  
 Perdue, H.S. - 179  
 Pergament, M.N. - 52  
 Prebluda, H.J. - 224  
 Prückner, H.E. - 340  
 Rabinovitch, H. - 292  
 Rapport, A. - 152  
 Reynolds, J.B. - 264  
 Reynolds, W.M. - 124, 133  
 Rice, E.E. - 52  
 Robeson, C.D. - 56  
 Rogerson, W.E. - 66  
 Rondenet, E.L. - 5  
 Rosenberg, A. - 57, 59, 63, 161, 162  
 Rosenberg, H.R. - 189  
 Rosenthal, W. - 246  
 Rosner, L. - 98  
 Ross, E.J. - 291  
 Rothschild, D.E. - 327  
 Rowoth, O.A. - 216  
 Rubin, M. - 38  
 St. Clair, G. - 130  
 St. Hilaire, R. - 293  
 Sauer, F. - 103  
 Schanze, R.F.M. - 340  
 Schara, R.E. - 280



## Inventor Index

- |                            |                              |
|----------------------------|------------------------------|
| Schmidt, J.L. - 179        | Tribble, T.B. - 5, 82        |
| Schmidt, P. - 205          | Tshudy, D.R. - 24            |
| Schöner, S. - 181          | Tucker, E.J., Jr. - 200      |
| Schoolmeister, J.E. - 208  | Tung, C.C. - 146             |
| Schumacher, E. - 205       | Ukita, T. - 114              |
| Schuppon, C.R.M. - 129     | Upham, S.D. - 116            |
| Sheneman, J.M. - 2         | Van Dijck, P. - 128          |
| Sherman, W.C. - 86, 133    | van Leeuwen, P.H. - 222      |
| Sicilia, E.G. - 243        | van Tittelboom, M.L.E. - 340 |
| Silbert, N.E. - 60         | Vasilakes, G.S. - 303        |
| Simmons, J.R. - 152        | Visek, W.J. - 213            |
| Simonet, J.F. - 15         | Wachtstetter, J.E. - 217     |
| Singer, P.A. - 254         | Walldov, G.A. - 294          |
| Sipos, E. - 50             | Wallhäuser, K.H. - 131       |
| Smith, F.H. - 43           | Ward, G.E. - 110, 142        |
| Snyder, J.M. - 216         | Webb, C.S. - 151             |
| Speer, P. - 312            | Weber, G.R. - 74             |
| Speer, V.C. - 278          | Wehrmeister, H.L. - 89, 139  |
| Stanton, N.K. - 84         | Weisberg, S.M. - 160         |
| Stelzner, H.D. - 197       | Wenger, L. - 39              |
| Stewart, C.W. - 226        | Wenger, L.G. - 39            |
| Stob, M. - 87              | Werblud, M. - 307            |
| Stokstad, E.L.R. - 127     | Wertz, P.F. - 224            |
| Streiff, K. - 223          | Wiley, J.R. - 185            |
| Tabenkin, B. - 275         | Wilhelm, M. - 205            |
| Tardani, A. - 140          | Wilkening, M.C. - 268        |
| Taylor, H.M. - 172         | Williams, L.S. - 336         |
| Teichman, R. - 183         | Williams, M.A. - 288         |
| Templeton, R.A.S. - 257    | Williams, W.L. - 121         |
| Terase, T. - 332           | Wineman, R.J. - 207          |
| Thompson, J.E. - 10, 321   | Winn, R.M. - 65              |
| Thompson, P.A. - 111       | Witte, N.H. - 50             |
| Thompson, R.Q. - 247       | Yakstis, S. - 298            |
| Thrasher, G.W. - 237       | Yamazaki, T. - 114           |
| Thurman, B.H. - 44, 48, 51 | Young, H.H. - 290            |
| Titus, H.W. - 259          | Ziffer, J. - 108, 131        |
| Tominaga, S. - 332         | Zorn, R.A. - 106, 107        |

## U.S. PATENT NUMBER INDEX

|           |     |           |     |           |     |
|-----------|-----|-----------|-----|-----------|-----|
| 2,746,864 | 50  | 2,943,938 | 86  | 2,997,393 | 179 |
| 2,835,584 | 57  | 2,944,903 | 16  | 2,999,752 | 151 |
| 2,838,553 | 43  | 2,945,764 | 64  | 3,000,742 | 323 |
| 2,841,495 | 226 | 2,945,765 | 216 | 3,001,874 | 207 |
| 2,874,048 | 294 | 2,946,685 | 291 | 3,004,852 | 327 |
| 2,876,242 | 44  | 2,947,632 | 71  | 3,010,828 | 52  |
| 2,921,853 | 84  | 2,949,362 | 18  | 3,011,891 | 147 |
| 2,924,522 | 35  | 2,951,759 | 103 | 3,011,892 | 63  |
| 2,924,523 | 273 | 2,951,760 | 124 | 3,013,880 | 29  |
| 2,924,524 | 274 | 2,952,540 | 68  | 3,014,800 | 55  |
| 2,924,525 | 225 | 2,952,541 | 12  | 3,015,563 | 59  |
| 2,924,526 | 236 | 2,960,406 | 152 | 3,015,564 | 212 |
| 2,924,527 | 237 | 2,960,407 | 95  | 3,017,272 | 128 |
| 2,925,341 | 165 | 2,962,378 | 121 | 3,017,273 | 333 |
| 2,925,342 | 133 | 2,965,488 | 261 | 3,019,109 | 117 |
| 2,926,084 | 277 | 2,965,489 | 334 | 3,020,157 | 77  |
| 2,926,085 | 270 | 2,967,106 | 66  | 3,020,158 | 79  |
| 2,927,027 | 171 | 2,968,559 | 48  | 3,020,159 | 225 |
| 2,927,859 | 206 | 2,970,053 | 134 | 3,021,216 | 246 |
| 2,928,738 | 53  | 2,970,910 | 51  | 3,021,217 | 107 |
| 2,928,739 | 154 | 2,971,843 | 257 | 3,023,105 | 116 |
| 2,929,711 | 127 | 2,973,265 | 150 | 3,032,415 | 307 |
| 2,929,712 | 122 | 2,973,266 | 162 | 3,033,683 | 50  |
| 2,930,695 | 98  | 2,974,043 | 209 | 3,033,684 | 65  |
| 2,931,726 | 240 | 2,977,230 | 99  | 3,033,685 | 2   |
| 2,932,571 | 82  | 2,978,326 | 55  | 3,035,919 | 108 |
| 2,933,392 | 160 | 2,985,533 | 106 | 3,035,920 | 339 |
| 2,937,091 | 161 | 2,985,534 | 107 | 3,036,917 | 101 |
| 2,938,794 | 242 | 2,986,468 | 204 | 3,038,806 | 170 |
| 2,939,790 | 47  | 2,987,398 | 94  | 3,039,874 | 129 |
| 2,940,856 | 223 | 2,988,448 | 194 | 3,039,875 | 190 |
| 2,940,857 | 9   | 2,988,449 | 193 | 3,041,173 | 100 |
| 2,940,858 | 19  | 2,990,283 | 179 | 3,042,525 | 103 |
| 2,941,884 | 275 | 2,991,178 | 48  | 3,044,877 | 33  |
| 2,942,976 | 30  | 2,995,445 | 26  | 3,050,396 | 17  |
| 2,942,977 | 111 | 2,996,383 | 316 | 3,051,571 | 52  |



# U.S. Patent Number Index

|           |     |           |     |           |     |
|-----------|-----|-----------|-----|-----------|-----|
| 3,051,572 | 82  | 3,185,573 | 126 | 3,272,633 | 335 |
| 3,051,573 | 191 | 3,192,047 | 310 | 3,275,446 | 300 |
| 3,063,839 | 15  | 3,196,018 | 233 | 3,278,308 | 143 |
| 3,066,024 | 336 | 3,196,019 | 87  | 3,279,921 | 144 |
| 3,069,269 | 38  | 3,198,635 | 36  | 3,279,922 | 144 |
| 3,071,468 | 308 | 3,202,514 | 287 | 3,279,923 | 131 |
| 3,074,795 | 109 | 3,203,806 | 290 | 3,284,209 | 68  |
| 3,077,404 | 95  | 3,208,852 | 230 | 3,284,210 | 189 |
| 3,079,260 | 232 | 3,208,853 | 228 | 3,284,211 | 288 |
| 3,079,261 | 231 | 3,210,194 | 250 | 3,284,212 | 5   |
| 3,080,234 | 210 | 3,212,901 | 56  | 3,285,748 | 115 |
| 3,086,864 | 213 | 3,215,539 | 301 | 3,293,039 | 293 |
| 3,087,819 | 34  | 3,218,171 | 172 | 3,294,543 | 148 |
| 3,087,820 | 34  | 3,218,172 | 13  | 3,295,983 | 301 |
| 3,089,771 | 204 | 3,219,453 | 237 | 3,295,984 | 263 |
| 3,092,496 | 168 | 3,222,179 | 181 | 3,301,681 | 326 |
| 3,098,745 | 247 | 3,239,341 | 89  | 3,322,544 | 298 |
| 3,099,561 | 321 | 3,239,348 | 90  | 3,325,288 | 146 |
| 3,115,409 | 283 | 3,239,349 | 91  | 3,325,289 | 76  |
| 3,117,866 | 60  | 3,240,605 | 138 | 3,328,168 | 238 |
| 3,117,867 | 149 | 3,243,299 | 243 | 3,328,169 | 234 |
| 3,118,768 | 341 | 3,244,527 | 19  | 3,328,170 | 300 |
| 3,119,691 | 280 | 3,245,797 | 135 | 3,332,778 | 268 |
| 3,119,692 | 197 | 3,246,989 | 11  | 3,333,962 | 224 |
| 3,121,634 | 208 | 3,248,222 | 251 | 3,336,136 | 262 |
| 3,130,054 | 329 | 3,248,223 | 176 | 3,336,137 | 254 |
| 3,131,064 | 21  | 3,248,224 | 71  | 3,338,718 | 178 |
| 3,134,676 | 180 | 3,249,441 | 264 | 3,340,065 | 340 |
| 3,136,640 | 292 | 3,250,622 | 258 | 3,345,178 | 110 |
| 3,139,342 | 284 | 3,250,623 | 130 | 3,347,677 | 145 |
| 3,144,337 | 101 | 3,252,802 | 274 | 3,352,683 | 205 |
| 3,147,120 | 166 | 3,255,014 | 230 | 3,352,684 | 217 |
| 3,147,121 | 7   | 3,256,093 | 292 | 3,352,685 | 317 |
| 3,148,068 | 246 | 3,256,094 | 278 | 3,356,504 | 97  |
| 3,148,988 | 137 | 3,256,095 | 266 | 3,357,835 | 27  |
| 3,150,979 | 241 | 3,256,096 | 200 | 3,359,115 | 329 |
| 3,151,983 | 195 | 3,257,209 | 201 | 3,361,566 | 298 |
| 3,155,520 | 131 | 3,257,210 | 221 | 3,368,901 | 10  |
| 3,155,521 | 142 | 3,259,500 | 271 | 3,370,953 | 215 |
| 3,157,511 | 187 | 3,261,687 | 136 | 3,370,954 | 314 |
| 3,157,512 | 118 | 3,261,688 | 113 | 3,372,032 | 340 |
| 3,165,413 | 74  | 3,264,111 | 185 | 3,372,033 | 222 |
| 3,171,745 | 167 | 3,264,112 | 173 | 3,373,024 | 92  |
| 3,172,764 | 10  | 3,266,901 | 140 | 3,375,116 | 319 |
| 3,173,792 | 40  | 3,268,336 | 303 | 3,380,832 | 281 |
| 3,180,735 | 259 | 3,271,159 | 184 | 3,385,709 | 39  |
| 3,184,314 | 3   | 3,271,161 | 199 | 3,395,019 | 66  |
| 3,185,572 | 245 | 3,272,632 | 312 | 3,397,990 | 182 |

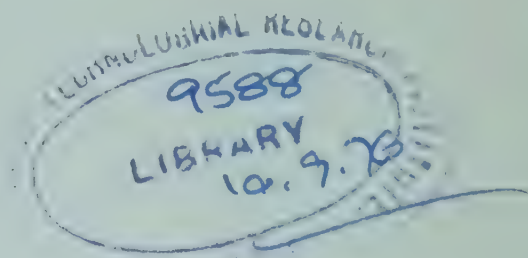
# U.S. Patent Number Index

|           |     |           |     |           |     |
|-----------|-----|-----------|-----|-----------|-----|
| 3,401,039 | 169 | 3,428,457 | 270 | 3,438,781 | 162 |
| 3,408,200 | 183 | 3,428,458 | 40  | 3,438,782 | 60  |
| 3,410,690 | 211 | 3,429,710 | 330 | 3,438,783 | 219 |
| 3,416,928 | 265 | 3,433,641 | 175 | 3,450,537 | 318 |
| 3,420,671 | 24  | 3,434,844 | 120 | 3,455,696 | 114 |
| 3,420,672 | 73  | 3,434,845 | 202 | 3,457,081 | 4   |
| 3,421,897 | 152 | 3,436,223 | 139 | 3,459,554 | 7   |
| 3,421,898 | 239 | 3,437,489 | 332 | 3,464,824 | 157 |
| 3,427,166 | 119 | 3,438,780 | 254 |           |     |



## NOTICE

Nothing contained in this Review shall be construed to constitute a permission or recommendation to practice any invention covered by any patent without a license from the patent owners. Further, neither the author nor the publisher assumes any liability with respect to the use of, or for damages resulting from the use of, any information, apparatus, method or process described in this Review.













# PURE CHEMICAL ELEMENTS FOR SEMICONDUCTORS 1969

by Marshall Sittig

*Electronics Materials Review No. 1*

This book gives detailed descriptive manufacturing techniques for producing pure chemical elements for use in semiconductors. In order to give up-to-date information, the book is based on the U.S. patent literature since 1960.

Dating from the discovery of the transistor in 1948 at the Bell Telephone Laboratories, there has developed a need for a whole new family of hyperpure materials. Any impurities in these materials are measured in parts per million or even in parts per billion.

The pure chemical elements discussed here may be used in elemental semiconductors—as in the case of silicon or germanium—or they may be components of compound semiconductors as in the case of gallium and arsenic in gallium arsenide, for example.

A group of new processes have been developed for the production of these hyperpurity materials involving the reduction of oxides and

halides, the disproportionation of halides, the decomposition of metal alkyls, the decomposition of metal hydrides, as well as various electrolytic techniques.

Following the various preparative methods listed above, special purification techniques are often used to attain the high purities required. These techniques include distillation, recrystallization, electrolytic refining, chemical treatment and zone refining.

Due to rapid commercial growth, some of these materials, particularly silicon are in short supply. Demand is growing rapidly and offers an interesting and profitable market for those alert chemical firms looking for new ventures.

The wide scope of detailed information provided by this book is shown in the Table of Contents outlined below:

## INTRODUCTION

### ANTIMONY

- Electrolytic Process
- Les Produits Semi-Conducteurs
- Chemical Reduction Processes
- Siemens-Schuckertwerke AG

### ARSENIC

- Arsenic Trioxide Purification
- U.S. Army
- Arsenic Trioxide Reduction
- Metropolitan-Vickers
- Arsenic Trichloride Reduction
- Texas Instruments

### BISMUTH

- Manufacture of Bismuth Whiskers
- General Mills
- Purification of Bismuth by Distillation
- Les Produits Semi-Conducteurs

### BORON

- Production from Boron Halides and Hydrogen
- Siemens & Halske AG
- Production from Boron Halides and Boron Hydrides
- General Electric Co.
- Purification by Heating Under Vacuum
- U.S. Borax & Chemical Corp.
- Purification by Zone Melting
- Bell Telephone Laboratories

### GALLIUM

- Production of Pure Gallium Halides for Electrolysis
- Pechiney
- Gallium by Electrolytic Precipitation from  $\text{Ga}(\text{GaX}_4)$  Solutions
- Siemens-Schuckertwerke AG
- Gallium by Electrolytic Precipitation from  $\text{Ga}(\text{AlBr}_4)$  Solutions
- Siemens-Schuckertwerke AG
- Production by Electrolysis of Acid Gallium Halide Solutions
- Monsanto
- Gallium by Disproportionation of  $\text{GaCl}_2$
- Radio Corp. of America
- Siemens-Schuckertwerke AG
- Gallium Purification by Decomposition of Gallium Alkyls
- Brown, Boveri & Co.
- Gallium Purification by Recrystallization from Melt
- Texas Instruments
- Gallium Purification by Molten Bath Electrolysis
- Siemens-Schuckertwerke AG

### GERMANIUM

- Purification of  $\text{GeCl}_4$  as a Raw Material for Pure Germanium
- American Metal Climax
- Purification of Germanium by Reduction of Germanium Dioxide
- American Metal Climax

### Production of Germanium by Decomposition of $\text{Ge}(\text{C}_2\text{H}_5)_4$

- Melpar
- Production of Germanium by Disproportionation of  $\text{GeI}_2$
- International Business Machines Corp.
- Production of High Density Germanium
- General Electric Co.
- Recovery and Purification of Germanium from Scrap
- Sylvania
- Germanium Purification by Zone Melting
- North American Philips
- Motorola, Inc.
- Germanium Decontamination by Out-Diffusion
- Motorola, Inc.
- International Business Machines Corp.
- Purification of Germanium by Electrolytic Refining
- General Trustee Co.
- Purification of Germanium by Double Electrolysis
- General Trustee Co.

### INDIUM

- Production by Electrolysis of Aqueous Solutions
- Duisburger Kupferhütte
- Les Produits Semi-Conducteurs
- Alloys Unlimited, Inc.

### PHOSPHORUS

- Purification by Acid Treatment and Steam Distillation
- Siemens-Schuckertwerke AG
- Purification by Zone Refining
- Knapsack-Griesheim AG

### SELENIUM

- Purification of Selenium by Hydrogenation
- Farbenfabriken Bayer AG

### SILICON

- Production of Silane as an Intermediate for Silicon
- Plessey Co.
- Du Pont
- International Standard Electric Corp.
- Purification of Silane as an Intermediate for Silicon
- International Standard Electric Corp.
- Production of Trichlorosilane as an Intermediate for Silicon
- Pechiney
- Purification of  $\text{SiHCl}_3$  as an Intermediate for Silicon
- Licentia Patent-Verwaltungs-GmbH
- Siemens & Halske AG
- Production of  $\text{SiCl}_4$  as an Intermediate for Silicon
- Purification of  $\text{SiCl}_4$  as an Intermediate for Silicon
- Bell Telephone Laboratories
- Production of Silicon by Thermal Decomposition of Silane
- St. Gobain
- Plessey Co.
- Merck
- Ishizuka
- International Standard Electric Corp.

### Feed Preparation

- Vapor Phase Process
- Melt Phase Process
- Production of Silicon by Pyrolysis of Trichlorosilane
- Wacker-Chemie
- Tung-Sol Electric
- Production of Silicon by Hydrogenolysis of Silicon Chlorides
- Westinghouse
- Sylvania
- Du Pont
- Aries
- Texas Instruments
- Siemens-Schuckertwerke AG
- Siemens & Halske AG
- Production of Silicon by Hydrogenolysis of  $\text{SiH}_4$
- U.S. Air Force
- Production of Silicon by Thermal Decomposition of  $\text{SiH}_4$
- Raytheon
- Mallinckrodt
- U.S. Air Force
- Du Pont
- General Electric
- Production of Silicon by Reduction of  $\text{SiCl}_4$  with Zinc
- Du Pont
- International Standard Electric Corp.
- Production of Silicon by Disproportionation of  $\text{SiX}_2$
- W. C. Heraeus
- Production of Silicon by Decomposition of Silicon Nitride
- U.S. Navy
- Production of Silicon from Silicon Monoxide and Hydrogen
- R.S. Aries
- Purification of Silicon by Recrystallization
- Wacker-Chemie
- Societe d'Ugine
- Union Carbide
- Purification of Silicon by Vacuum Distillation
- American Metal Products
- Melt Purification of Silicon
- Du Pont
- Dow-Corning

### TELLURIUM

- Production and Purification of Semiconductor Grade Tellurium
- Les Produits Semi-Conducteurs

### THALLIUM

- Production from Lead-Zinc Plant Residues
- Duisburger Kupferhütte

### ZINC

- Electrolytic Production of Hyperpure Zinc
- Montevecchio

### FUTURE TRENDS

### INDEXES

112 illustrations  
335 pages

**\$35**

# RADIATION CHEMICAL PROCESSING 1969

by R. Whiting

*Chemical Processing Review No. 41*

A number of radiation induced chemical processes are already operating commercially. The radiation processing of chemicals has been growing at an annual rate of about 25% per year. Currently, \$100 to 150 million worth of irradiated products are produced in the United States per year, however, it has been forecast that by 1980, the value of products receiving radiation treatment will be close to \$1,000 million per year.

Current applications include polymerization, cross-linking polymers, curing surface coatings, synthesis of ethyl bromide, production of wood-plastic combinations, controlled degradation of polymers, textile treatment, etc. There is an extensive variety of commercial processes, but more important, there are numerous newly developed processes that should have a significant technological impact.

As these new processes are put on-stream, there will be developed a quickened interest on the part of research and development management to explore radiation chemical processing more thoroughly. In its infancy, radiation was oversold as a solution to all problems. Today, a more intelligent approach is now developing new products and lowering production costs.

This book surveys the radiation processing field and is based on the U.S. patent literature since 1960. Over 250 separate processes are described in detail in the chemical, polymer, rubber, petroleum, textile and other fields. An outline of the Table of Contents is shown below. The numbers in ( ) indicate the number of processes described under that particular heading. This is a down-to-earth practical book of manufacturing technology.

## CONTENTS

### POLYOLEFINS

- Polymerization of Ethylene (7)
- Cross-Linking of Polyethylene (7)
- Modifying Properties of Polyethylene (12)
- Miscellaneous Polyethylene Compositions (7)
- Cross-Linking of Polypropylene (3)
- Modifying Properties of Polyolefins (4)
- Graft Polymers (3)

### OTHER POLYMERS

- Acrylics (9)
- Aldehyde Polymers (5)
- Epoxides (2)
- Fluorinated Polymers (5)
- Polyamides (2)
- Polyethers (4)
- Polyesters (4)
- Polyoxymethylene (8)
- Styrene Polymers (9)
- Vinyl Polymers (14)
- Others (5)

### ELASTOMERS

- Styrene/Butadiene Rubbers (6)
- Isobutylenes (5)
- Butadiene and Isoprene (4)
- Butyl Rubber (3)

- Ethylene Elastomers (2)
- Others (6)

### HYDROCARBONS

- Alkylation (6)
- Isomerization (6)
- Catalyst Modification (8)
- Oxidation (3)
- Lubricants (12)
- Waxes and Resins (3)
- Other Processes (16)

### ORGANIC CHEMICALS

- Nitrogen-Containing (11)
- Sulfur-Containing (6)
- Halogen-Containing (11)
- Others (13)

### INORGANIC AND ORGANO-METALLIC COMPOUNDS

- Inorganics (4)
- Organo-Metallics (5)

### OTHER PROCESSES

- Textiles (7)
- Others (6)

### INDEXES

375 pages

**\$35**

This book is based on U.S. patent technology in the microencapsulation field. Detailed descriptions and illustrations of processes and products are given. An abbreviated table of contents is outlined below:

Phase Separation Methods; Coacervation-Aqueous Phase Separation, Organic Phase Separation, Spray Drying, Miscellaneous: Interface Reactions—Polymerization; Dissolved Monomer Polymerization, Interfacial Polymerization, In-situ Polymerization, Vapor Deposition: Physical Methods; Fluidized Bed, Electrostatic, Multi-Orifice Centrifugal, Vacuum Metallizing, Coating Fusible Material: Applications; Xerographic Toner, Light Sensitive Photographic Materials, Heat Sensitive Transfer Sheets—Dyes, Miscellaneous, Indexes. Illustrations. 275 pages. \$35

### SUGAR ESTERS 1968

Early History, Nebraska-Snell Process, Present Status of Processes, Sugar Outlook, Use of Sucrose Monopalmitate, Applications in Foods, Animal Feeding, Sucrose Esters in Cosmetics, Sucroglycerides in Foods, The Enhancement of Herbicide Sprays, Herbicide Adjuvants, Effectiveness of Fat-Sugar Derived Surfactants, Laundering Performance, Sucrose Ester Detergents, Evaluation of Sucrose Monotallowates, Water Quality, Water Pollution Problems. 134 pages. \$15

### COSMETICS INDUSTRY OF EUROPE 1968

by S. A. Mann

This report outlines the cosmetics industries in 19 European countries.

The report is particularly valuable in that it points out the differences in the various European countries as compared to the United States. The statistical material helps you evaluate the potential markets in each country. The lists of cosmetics and toiletry companies help you in your marketing efforts. 114 pages. \$35

### EUROPEAN MAN-MADE FIBER MARKET REPORT 1968

by S. A. Mann

This book will give you an overall picture of the man-made fiber situation in Western Europe. It is a valuable work that will help you in market research studies. Never before has so much statistical information on this subject been included between the covers of one publication.

In the chapters discussing individual countries there are numerous tables pertaining to production, consumption, exports and imports. 186 pages. \$35

### EUROPEAN PETROCHEMICAL FACTS 1968

by Alexander Erdelyi, Jr.

Contents: Analysis by product; Summary of supply/demand situations and forecasts for basic petrochemicals in Europe; Analysis by country; Summary of major companies and their product lines; product flow analysis.

For major European petrochemicals it gives plant locations, capacities, processes, feed suppliers, customers, trends, etc. Analyzes three ways—by product, country, and company. 176 pages. \$38



# ALCOHOLIC MALT BEVERAGES 1969

by M. Gutcho

*Food Processing Review No. 7*

Brewing is an industry where basic processes have not changed in over 100 years, it is complicated by the fact that it is the flavor of the product that is of primary importance, rather than the quantitative value of its components. Since it is a biochemical process it is also affected by variations in starting materials such as malt, hops and yeast.

The traditional brewing process is a batch operation, costly and time consuming. There would be economic advantages to improved continuous processes which would require less capital investment for plant and equipment, give savings in labor, better use of raw materials, shorter processing time, and a more uniform product.

Detailed descriptive process information is found in this review, based on 157 U.S. Patents in the brewing field, issued since 1960. The 157 processes are arranged in 7 chapters which tend to follow the steps in the brewing process.

The wide variety of these recent developments in this field is shown by the abbreviated Table of Contents outlined below. The numbers in ( ) indicate the number of processes described under that particular heading.

## MALTING

- Steeping Modifications (7)
- Treatment at Steep Out to Increase Malting Yields (4)
- Treatment at Steep Out to Increase Enzyme Activity (2)
- Increasing Growth and Enzyme Activity During Germination (1)
- Increasing Malting Yields During Germination (4)
- Grain Modification (1)
- Kilning (3)
- Continuous Malting Process (1)
- Malt Extract (1)

## WORT

- Filtration Improvement (4)
- Continuous Lautering Processes (11)
- Concentration of Wort (2)
- Continuous Boiling and Hopping (6)
- Specific Modification of Wort to Improve Final Beer (6)
- "Cooker" Mash Modifications (3)
- Nonconventional "Malt" Mash (4)
- Edible By-Product from Spent Grains and Yeast (1)

## HOPS

- Treatment of Hops Prior to Use (2)
- Lupulin (4)
- Hop Extracts and Concentrates (9)
- Light Stable Beverages Via Reduction of Hop Extracts (3)

## FERMENTATION

- Batch Processes (5)
- Continuous Processes (9)
- Inhibiting (2)
- Fermentation Modifications to Achieve Specific End (7)
- Lagering (4)
- Carbonating (1)

## FREEZE CONCENTRATION AND RECONSTITUTION OF BEER (8)

## CHILLPROOFING

- Adsorbent Silicates and Silica Gel (4)
- Natural Clays as Adsorbents (5)
- Specific Polymeric Adsorbents (3)
- Polymeric as Stabilizers (2)
- Nonproteolytic Enzymes (3)
- Animal Blood Albumin as Coagulant and Precipitant (1)
- Antioxidants (2)
- Chelating Agents (2)

## PRESERVATION AGAINST MICROBIOLOGICAL SPOILAGE

- Chemical Preservatives (7)
- Pasteurization (2)
- Cleanliness (1)

## FOAM

- Foam Stabilizers (6)
- Cling (5)
- Gushing (1)

## INDEXES

333 pages

**\$35**

**Noyes Development Corporation—**

Noyes Building—Park Ridge, New Jersey 07656—U.S.A.

## PHOTOCHEMICAL PROCESSES 1969

by B. Albertson

*Chemical Processing Review No. 36*

The purpose of this Review is to provide an up-to-date description of industrial photochemical technology as recorded in the U.S. Patent literature since 1960. Describes in detail 210 photochemical production processes.

This book describes a wide variety of processes to produce specific organic chemicals and polymers. A few inorganic photochemical processes are also included.

Photochemistry, long known as a selective powerful tool, has excited the imagination of chemical engineers as a method of manufacturing various chemicals. From the economic point of view, photochemistry has the following advantages: 1. Many materials cannot be obtained in any other way. 2. Specificity of effects cannot be obtained by other methods. 3. Less need for high pressures and temperatures can lower costs.

Introduction, Photohalogenation, Photonitrosation, Organic Photochemical Reactions, Inorganic Photochemical Reactions, Photopolymerization, Indexes. Illustrations. 185 pages. \$35

## EUROPEAN AGRICULTURAL CHEMICAL SURVEY 1969

by S. A. Mann

This is a guide to the European Agricultural Chemicals Industry that will help you in sales, market research, development, planning, acquisitions and mergers. The pesticides considered in this survey include all plant or crop protection chemicals such as insecticides, herbicides, fungicides, etc.

There are three types of information presented for each country: 1. Discussion of the industry, including market structure. 2. Statistical material on production, consumption, imports, and exports. 3. The names and addresses of the leading pesticide producers in each country. This book contains a total of about 1,000 names and addresses of Europe's leading pesticide producers.

This book contains considerable statistical material, invaluable for market research studies. Much of the statistical material is broken down in considerable detail. 129 pages. \$35

## GUIDE TO CHEMICAL PLANT PLANNING 1969

by Dr. Robert Lobstein

The assemblage of information needed for selecting materials and equipment, and for fitting them into the dimensional scheme, can present a major problem to the most experienced of engineers and architects. Endless perusal of catalogs, technical magazines, and handbooks often not specifically concerned with the problem at hand becomes necessary. One purpose of this book is to make much of the searching effort unnecessary. The required information has been collected and condensed into clear, quickly available essential data. It has been unified.

Much of the book is intended as an aid in the selection of mechanical equipment. The practical approach must include an understanding of machinery requirements as well as practical physics and chemistry. Many tables and diagrams in current usage by equipment and machinery suppliers are included.

Contents: Introduction, Piping, Pumps, Heat, Power Transmission, Mechanical Equipment, Electrical Engineering, Operational Requisites. 452 charts. 524 pages. \$24

## CHEMICAL GUIDE TO EUROPE 1968

Fourth Edition

Describes the 1,000 leading European chemical manufacturers in 18 countries with this information (where available):

Name and Address, Ownership, Plant Locations and Products, Local Subsidiaries and Affiliates, Foreign Subsidiaries and Affiliates, Principal Executives, Annual Sales, Number of Employees.

A valuable marketing guide. Only the 1,000 major chemical firms are included. This allows you to concentrate your efforts in the most profitable direction.

These are the firms that have the most to offer in the way of sales contacts, licensing arrangements, joint ventures, and the obtaining of research and development know-how. In order to give you an indication of the size of each firm we have included, where available, the annual sales figure and/or the number of employees. The European chemical industry is almost as large as that of the United States and is too important to ignore. Every chemical executive should be aware of how the European chemical industry is organized. 207 pages. \$20

## FIBRINOLYTIC ENZYME MANUFACTURE 1969

by T. Rubel

*Chemical Processing Review No. 38*

The formation of blood clots in the human body is a frequent cause of death. Standard anti-coagulant drugs are not effective in removing these clots. The proteolytic enzymes have shown great promise in clot lysis. The new enzyme urokinase appears particularly interesting due to no bad side effects, and few changes in blood composition.

One of the elements taking part in clot lysis is present in blood in the form of a normally inactive factor called profibrinolysin or plasminogen which, when activated, forms fibrinolysin or plasmin which acts upon the proteinaceous fibrin to break it down, thereby lysing the clot.

This Review presents some methods for the production and purification of fibrinolytic agents, their precursors and activators. Particular emphasis is on the enzymes urokinase and streptokinase which activate plasminogen to form plasmin.

Plasminogen and Fibrinolysin, Urokinase, Streptokinase and Streptodornase, Other Fibrinolytic Enzymes. Indexes. 139 pages. \$24

## POLYMETHYLBENZENES 1969

by H. W. Earhart

This book presents detailed physical property data on the polymethylbenzenes (PMB's) and their derivatives, discusses the chemistry of the PMB's, and presents known as well as suggested end-uses for numerous PMB derivatives in the fiber, plastic, coatings, rubber, dye, medical, agricultural, and other industries.

The purpose of this book is to present a comprehensive compilation of technical information concerning the eleven polymethylbenzenes: Benzene, Toluene, Xylene, Mesitylene, Pseudocumene, Hemimellitene, Durene, Isodurene, Prehnitene, Pentamethylbenzene, Hexamethylbenzene.

Complete physical property data, established chemical reactions giving relative kinetic rate data, yields, etc. and end-use applications are presented and discussed in detail. Other related information is also presented.

This book contains a substantial amount of data on this subject. There are 63 detailed tables. The bibliography is extensive and includes 549 literature references. \$20



# **BASIC PATENTS FOR MAJOR DRUGS 1969**

This book contains the basic manufacturing patents for the 301 major drugs most frequently prescribed in the United States. The patents are organized by generic name in three separate bindings totaling 1705 pages. The actual patents themselves are reproduced in this book to insure accurate transmittal of information.

The book actually contains 341 U.S. Patents, since for some of these drugs there is more than one basic patent. Also, certain patents cover more than one drug.

A valuable feature of this book is that you can easily obtain patent expiration dates. A separate chronological and patent number index lists all of the drugs in this book by patent issue date and it is simple to determine what drugs are "out-of-patent" at any point in time. Or, more important, it is simple to determine what drugs will be "out-of-patent" in the near future. A therapeutic index is also included. 1705 pages. 3 bindings. \$75

# **SUSTAINED RELEASE PHARMACEUTICALS 1969** by Alec Williams

This book is based on the U.S. patent literature, and presents substantial technical information for production of these products.

An abbreviated table of contents is outlined below.

## **TABLETS-PILLS**

Cellulosic Coatings  
Lipid Coating  
Gels-Gums  
Specific Medicaments  
Tablet Design  
Miscellaneous

## **CAPSULES**

General Coatings  
Particle Size Control  
Capsule Design & Manufacture

## **INJECTABLES**

Antibiotics  
Miscellaneous

## **MISCELLANEOUS RELEASE PREPARATIONS**

Suppositories  
Powders  
General

Indexes. 273 pages. \$35

# **COMBINE HYDROCARBONS AND NITROGEN FOR PROFIT 1967**

by Marshall Sittig

*Chemical Process Review No. 8*

Contents: Introduction, Products From Ammonia, Diethylamine and its Derivatives, Products from Nitric Acid, Alkanolamines and their Derivatives, Pyridine and its Derivatives, Urea Derivatives, Nicotinic Acid and its Derivatives, Products from Calcium Cyanamide, Caprolactam and its Derivatives, Ethylene Imine and its Derivatives, Future Trends. 64 illustrations. 200 pages. \$35

# **TEXTURED AND NOVELTY YARN PROCESSES 1967**

by Mark Harrison

In the world today, hundreds of millions of pounds of synthetic fibers are being textured by a wide variety of processes. Yet, for those of you who wish to explore this important subject, there is very little published material.

Chapters: Introduction, Classification, Compressive Stressing, Torsional Stressing, Tensile Stressing, Tensile and Compressive Stressing (Combinations). 315 pages and many illustrations. \$35

# **COMBINE HYDROCARBONS AND HALOGENS FOR PROFIT 1968**

by Marshall Sittig

*Chemical Process Review No. 10*

Contents: Introduction, Production of Halogenated Methane Derivatives, Production of Halogenated Ethanes and Ethylenes, Production of Halogen Derivatives from Propylene, Production of Halogen Derivatives of Butadiene, Production of Halogen Derivatives of Higher Paraffins, Production of Halogenated Aromatics, Future Trends. 62 illustrations. 181 pages. \$35

# **STRATEGY FOR PATENT PROFITS 1967**

by Roland Dori

Chapters: Patents and Their Creation; Trade Secrets, Infringements and Shop Rights; The Licensing and Assignments of Patents; The Difficulties in Marketing a Patent; The Influence of Technological Change on Patent Utilization; Patent Development and Marketing Channels; Methods of Developing a Market for Patents; The Activities of Patent Brokers; Aspects of Patent Economics; Corporations and Their Patent Activity; Case Histories. Bibliography. 138 pages. \$18

# **CHLORINE AND CAUSTIC SODA MANUFACTURE RECENT DEVELOPMENTS 1969**

by Dr. Robert Powell

*Chemical Process Review No. 33*

Discusses up-to-date manufacturing techniques for chlorine and caustic soda. This book reflects these latest developments. It includes such subject matter as brine preparation, electrolysis in diaphragm and mercury cells and other electrolysis techniques, catalytic oxidation, nitrosyl chloride route, cooling, drying and purification. 265 pages. \$35

# **COMBINE HYDROCARBONS AND OXYGEN FOR PROFIT 1968**

by Marshall Sittig

*Chemical Process Review No. 11*

Contents: Introduction; Cyclic Oxides from Olefins and Diolefins; Acids, Anhydrides and Esters; Olefins and Diolefins by Oxidehydrogenation; Aldehydes and Ketones from Olefins; Alcohols from Paraffins and Cycloparaffins; Peroxy Compounds; Future Trends. 214 pages. \$35

# **ACETYLENE PROCESSES AND PRODUCTS 1968**

by Marshall Sittig

*Chemical Process Review No. 22*

Describes specific processes for acetylene production, and acetylene derived products.

Contents: Introduction, Manufacture of Acetylene. Separation of Acetylene from Gas Mixtures, Handling of Acetylene, Reactions of Acetylene, Future Trends. 90 illustrations. 214 pages. \$35

# **SYNTHETIC FIBERS FROM PETROLEUM 1967**

by Marshall Sittig

*Chemical Process Review No. 1*

This book discusses production processes for nylons, polyesters, acrylics, and polyolefins. The trend in the industry has been to modify fibers specifically for certain uses and then to protect the modification by patents.

This process review evaluates technically the synthetic fiber industry. A wealth of processing facts. 116 illustrations. 275 pages. \$35

# **MONOSODIUM GLUTAMATE AND GLUTAMIC ACID 1968**

by Dr. Robert Powell

*Chemical Process Review No. 25*

Hydrolysis of Vegetable Proteins, Recovery of Glutamic Acid, Biosynthesis from Carbohydrates, Ketoglutaric Acid, Hydrocarbons and other Materials, Recovery of Glutamic Acid from the Fermentation Broth, Chemical Synthesis of Glutamic Acid, Chemical Synthesis of Intermediates, Optical Resolution Methods, Preparation and Recovery of MSG. Numerous illustrations. 256 pages. \$35

# **AROMATICS MANUFACTURE AND DERIVATIVES 1968**

by Marshall Sittig

*Chemical Process Review No. 17*

A summary of production techniques for aromatics and their derivatives.

Contents: Introduction, Production of Aromatics, Separation of Aromatics, Purification of Aromatics, Reactions giving Hydrocarbon Products, Other Reactions, Phenol Production, Styrene Manufacture and Derivatives, Future Trends. 73 illustrations. 232 pages. \$35

# **CATALYSTS AND CATALYTIC PROCESSES 1967**

by Marshall Sittig

*Chemical Process Review No. 7*

In this book, catalysts are discussed in terms of the processes which use the catalysts. In each case major emphasis is placed on the catalyst itself.

Contents: Hydrocarbons Conversion Processes, Hydrocarbon Polymerization Processes, Hydrocarbon Oxidation Processes. Future Trends. 109 illustrations. 303 pages. \$35

# **PLASTIC FILMS FROM PETROLEUM RAW MATERIALS 1967**

by Marshall Sittig

*Chemical Process Review No. 6*

In this field there is a great scarcity of practical information dealing with the materials, equipment, techniques and operating conditions used in actual manufacturing processes. This Review will overcome that need and introduce the reader to the practicalities of polymer manufacture, film production, and film treatment processes. 118 illustrations. 276 pages. \$35

# **OLEFINS-MANUFACTURE AND DERIVATIVES 1968**

by Marshall Sittig

*Chemical Process Review No. 12*

This is a technological review of olefins—manufacture, separation, purification, and reactions. Oriented toward industrial practice, this Review gives you basic information relating to olefins.

Contents: Introduction, Manufacture of Olefins, Separation of Olefins, Purification of Olefins, Reaction of Olefins. Future Trends. 213 pages. \$35

# **POLYOLEFIN PROCESSES 1967**

by Marshall Sittig

*Chemical Process Review No. 2*

This book is devoted to manufacturing techniques and processes for polyolefin resins.

Includes: Current Status of Various Polyolefins, Processes Using Non-Metallic Catalysts, Processes Using Metal Oxide Catalysts, Processes Using Metal Alkyl-Reducible Metal Halide Catalysts, Polymer After-Treatment, Polymer Fabrication, Future Trends. 96 illustrations. 234 pages. \$35

# **COMPATIBILITY AND SOLUBILITY 1968**

by Ibert Mellan

This book helps you evaluate proper materials by the use of 224 tables. The book contains a wealth of information all conveniently grouped to help save you time and money. The tables are organized in three sections—polymers and resins, plasticizers and esters, and solvents. The tables in each section indicate the solubility and compatibility of the particular material with a wide range of other materials. 304 pages. \$20

# **LIQUEFIED NATURAL GAS TECHNOLOGY AND ECONOMICS 1967**

by C. H. Gatton

A wealth of technical and economic data. Includes these chapters: Liquefied Petroleum Gas, Marine Transportation of LPG, Liquefied Natural Gas, Liquefaction and Revaporization of LNG, LNG Storage, Marine Transportation of LNG, LNG Economics and Future Development, Marine Transportation of Other Liquefied Gases, Bibliography, Appendix—Economics of Sea Transport of LNG. 185 pages. \$15

# **DIOLEFINS—MANUFACTURE AND DERIVATIVES 1968**

by Marshall Sittig

*Chemical Process Review No. 10*

Available in huge quantity at low prices as elastomer raw materials, the diolefins offer attractive possibilities as petrochemical raw materials.

Contents: Introduction, Manufacture of Diolefins, Separation of Diolefins, Purification of Diolefins, Manufacture of "Big Ring" Compounds, Reactions of Diolefins, Future Trends. 61 illustrations. 197 pages. \$35

# **MEMBRANES TECHNOLOGY AND ECONOMICS 1967**

by Dr. Robert Rickles

This book presents both the technical and economic factors that govern the present and future prospects in this field. The status of membranes, equipment and processes are discussed.

Chapters: Membrane Theory, Electrodialysis, Ultrafiltration, Dialysis and Diffusion Control, Membrane Filtration, Gas Permeation, Medical Applications, Preparation of Synthetic Polymeric Membranes. 197 pages. \$24

# **PLASTICIZER EVALUATION AND PERFORMANCE 1967**

by Ibert Mellan

Will help you evaluate: 1. A new plasticizer with a known resin; 2. A known plasticizer with an unknown resin; 3. A new plasticizer with a new resin. Introduction, Testing, Comparative Performance, Performance Data, Brand Names and Manufacturers, Abbreviations for Coding, Chemical Names of Plasticizers and Brand Names. Bibliography. 178 pages. \$20

# **CHEMICAL GUIDE TO GATT, THE KENNEDY ROUND, AND INTERNATIONAL TRADE 1968**

by Yale Meltzer

This book will serve to guide the reader through the intricate labyrinth of intertwining issues connected with the Kennedy Round, the proposed SDR plan and devaluation of the British pound. These issues cover a vast array of practical R&D, technological, manufacturing, marketing, exporting, importing, financial, legal, fiscal and political problems. 379 pages. \$20



# SYNTHETIC LEATHER FROM PETROLEUM 1969

by Marshall Sittig  
*Chemical Process Review No. 29*

The newer types of synthetic leather offers a true leather substitute; far superior to older "artificial" leathers such as vinyl coated fabrics. The newer synthetic leathers offer permeability to water vapor and air, as does natural leather. In addition, the newer products offer good tear strength and softness. It is expected that these newer synthetic leathers will make substantial inroads in shoe and other markets.

These modern synthetic leathers consist, in general, of nonwoven mats of such fibers as polyesters impregnated with such binder resins as polyurethanes. The fiber-binder combination is then rendered porous by one of a number of different processes.

Introduction: Manufacture of Fiber-Forming Polymers, Types of Structural Fibers Used, Mat Formation, Mat Treatment, Binder Polymer Formulation, Other Ingredients, Fiber-Binder Combination, Consolidation, Coating, Introduction of Porosity, Surface Modification, Product Properties, Future Trends. 84 illustrations. 214 pages. \$35

# ALKALI METAL PHOSPHATES 1969

by Dr. M. W. Ranney  
*Chemical Processing Review No. 34*

This book describes recent processes for production of alkali metal phosphates. The 97 processes are based on U.S. patents issued since 1960 and offer a comprehensive treatment of up-to-date technical information.

Contents: Orthophosphates, Metaphosphates, Triphosphates, Phosphites/Hypophosphites.

Numerous illustrations. 344 pages. \$35

# CARBON BLACK TECHNOLOGY RECENT DEVELOPMENTS 1968

by Dr. Robert Powell  
*Chemical Process Review No. 21*

1. Introduction; 2. Feedstocks; 3. Channel Blacks; 4. Furnace Blacks; 5. Thermal Blacks; 6. Acetylene Blacks; 7. High Structure Carbon Blacks; 8. Low Structure Carbon Blacks; 9. Unconventional Processes; 10. Carbon Black Pelletizing; 11. Other Finishing Treatments. Numerous illustrations. 242 pages. \$35

# INDUSTRIAL GASES MANUFACTURE AND APPLICATIONS 1967

by Marshall Sittig  
*Chemical Process Review No. 4*

Discusses cryogenic air separation and purification techniques in detail. Plants have now reached a high state of refinement as regards low cost, efficient, safe operation. Many patents on separation processes have expired.

This is a down-to-earth practical book summarizing the present state of the art, including all new developments. 313 pages. 103 illustrations. \$35

# AMINES, NITRILES AND ISOCYANATES PROCESSES AND PRODUCTS 1969

by Marshall Sittig  
*Chemical Process Review No. 31*

This book describes processes for producing amines, nitriles, and isocyanates, the backbones of many important commercial polymeric materials.

Introduction: Manufacture of Amines, Manufacture of Mono-Nitriles, Manufacture of Mono-Nitriles, Acrylonitrile Derivatives, Isocyanate Manufacture, Future Trends. 62 illustrations. 201 pages. \$35

# TITANIUM DIOXIDE AND TITANIUM TETRACHLORIDE 1968

by Dr. Robert Powell  
*Chemical Process Review No. 18*

A technological review of titanium dioxide processes, based on chlorination of rutile ore. It describes the various processing steps (a) chlorination of the ore, (b) separation of sublimated solids, (c) purification of crude titanium tetrachloride, and (d) conversion of titanium tetrachloride to titanium dioxide. Numerous illustrations. 306 pages. \$35

# STEREO-RUBBER AND OTHER ELASTOMER PROCESSES 1967

by Marshall Sittig  
*Chemical Process Review No. 3*

The synthesis of new polymers having rubber-like elasticity is now a vital and growing industry. The further modification of the molecular architecture of such elastomers to impart specific properties, is the subject of world-wide research. This Review summarizes the present state of the art in the elastomer industry. It covers the field, including new routes. 215 pages. 77 illustrations. \$35

# FUEL CELLS RECENT DEVELOPMENTS 1969

by Dr. M. W. Ranney

This book describes in detail a wide range of recent developments in fuel cell technology, based on the latest U.S. Patents.

Fuels; Hydrocarbons, Nitrogen Compounds, Miscellaneous: Electrolytes; Solid Electrolytes, Electrolyte Additives, Liquid Electrolytes, High Temperature Electrolytes, Miscellaneous: Membranes-Separators; Ion-Exchange Membranes, Separators, Barriers: Electrodes; Catalysts, Membranes-Hydrophobic, Carbon Electrodes-Wetproofing, Control of Porosity, Miscellaneous Electrode Construction: Fuel Cell Construction; General, Control Systems, Water Removal-Control, Pressure Variation and Control: Biochemical and Thermocells: Biochemical Cells, Thermocells: Regeneration and Reactivation; Regeneration, Reactivation, Indexes. Many illustrations. 325 pages. \$35

# VITAMIN E MANUFACTURE 1969

by T. Rubel  
*Chemical Processing Review No. 39*

Tocopherols are complex alcohols found in natural fats and oils, particularly in vegetable oils, such as soybean oil, wheat germ oil or cottonseed oil. There are seven known tocopherols, the more common being alpha tocopherol, beta tocopherol, gamma tocopherol and delta tocopherol. Of these, alpha tocopherol exhibits the highest vitamin E biological activity. A deficiency of vitamin E may cause sterility, muscular dystrophy and cardiac lesions in some animals. Its exact therapeutic value to humans has not been definitely established.

This review relates the known methods for the preparation of tocopherols from natural products or by synthetic means, the conversion of non-alpha tocopherols to the more active form, and miscellaneous related processes.

Introduction: Tocopherols From Deodorizer Sludge, Conversions to Alpha Tocopherol, Synthesis of Tocopherols, Miscellaneous Related Processes, Indexes. 114 pages. \$24

# PARAFFINS AND CYCLOPARAFFINS MANUFACTURE AND DERIVATIVES 1968

by Marshall Sittig  
*Chemical Process Review No. 19*

The olefins are the firm backbone of the hydrocarbon-based industries.

Contents: Introduction, Production of Paraffins and Cycloparaffins, Separation of Paraffins and Cycloparaffins, Purification of Paraffins, Reactions of Paraffins, Reaction of Cycloparaffins. 92 illustrations. 234 pages. \$35

# HYDROGEN PEROXIDE MANUFACTURE 1968

by Dr. Robert Powell  
*Chemical Process Review No. 20*

Shows processes of such firms as FMC Corp., Air Liquide, CIL, Laporte, Ugine, Montecatini, PPG, duPont, Shell, Kali-chemie, etc.

Contents: General Considerations, The Anthraquinone Process, Other Processes, Purification, Concentration, Stabilization. Numerous illustrations. 221 pages. \$35

# ALCOHOLS, POLYOLS, AND PHENOLS MANUFACTURE AND DERIVATIVES 1968

by Marshall Sittig  
*Chemical Process Review No. 23*

A technological review of processes for alcohols, polyols, and phenols.

Contents: Introduction, Manufacture of Alcohols, Manufacture of Glycols, Manufacture of Polyols, Manufacture of Phenol, Reactions of Alcohols, Reactions of Glycols and Polyols, Phenol Derivatives. 71 illustrations. 201 pages. \$35

# HYDRAZINE MANUFACTURING PROCESSES 1968

by Dr. Robert Powell  
*Chemical Process Review No. 26*

General Considerations, Raschig Process, Bayer Process, Processes Based on Urea, Electrical Glow Discharge Processes, Chemo-Nuclear Processes, Direct Production of Anhydrous Hydrazine, Other Processes, Recovery, Concentration, Stabilization. Numerous illustrations. 252 pages. \$35

# DYEING OF SYNTHETIC FIBERS 1969

by C. Whiting  
*Textile Processing Review No. 1*

Water-soluble dyes are not easily applicable and the dyeing of synthetic fibers continues to be a challenge. Successful methods for dyeing these fibers, however, have been developed. Chapters 2 through 7 of this Review are divided into two sections. The first presents various new dyes and dyeing processes which are applicable to synthetic fibers. The second section concerns the many auxiliary products available to aid in production of an acceptably dyed product. These include, for example, dye improving agents, leveling and retarding agents, as well as agents used in after treatment to achieve the optimum fastness properties.

Introduction: Dyeing Polyolefin and Polypropylene Fibers, Dyeing Polyamide Fibers, Dyeing Polyester Fibers, Dyeing Acrylic Fibers, Dyeing Hydrophobic Fibers, Dyeing Glass Fibers, Dyeing Miscellaneous Fibers, Dyes Applicable to More than One Type of Fiber, Dyeing Fiber Blends. 257 pages. \$35

# PRACTICAL DETERGENT MANUFACTURE 1968

by Marshall Sittig  
*Chemical Process Review No. 27*

Introduction, Manufacture of Branched-Chain Olefins, Manufacture of Linear Alpha Olefins, Manufacture of Linear Paraffins, Competitive Routes to Straight-Chain Alcohols, Competitive Routes to Alkylaromatics, Routes to other Detergent Raw Materials, Sulfation and Sulfonation Processes, Detergent Formulation, Future Trends. 76 illustrations. 212 pages. \$35

# POLYACETAL RESINS, ALDEHYDES, AND KETONES 1968

by Marshall Sittig  
*Chemical Process Review No. 26*

The aldehydes and ketones can, in general, be classified as among the most important tonnage organic chemical intermediates and solvents.

Contents: Introduction, Production of Aldehydes, Production of Ketones, Reactions of Aldehydes, Reactions of Ketones, Aldehyde Polymers, Future Trends. 81 illustrations. 237 pages. \$35

# ELECTRO-ORGANIC CHEMICAL PROCESSING 1968

by Dr. Charles Mantell  
*Chemical Process Review No. 14*

This volume has been written from the viewpoint of the chemical engineer, with emphasis on plant processes, operating data, and plant design. The commercial successes in this field have been attained by the chemical engineering approach. An important factor in the recent success of electro-organic processes is the much greater variety of engineering materials now available. 186 pages. \$35

# CORROSION RESISTANT MATERIALS HANDBOOK 1966

by Ibert Mellan

The key to the value of this book is the extensive double indexing. The 95 Tables themselves are arranged by type of corrosion resistant material. The extensive Index is organized by corrosive chemical—and refers you to the specific recommendations in the Tables.

If you have a problem with a corrosive chemical, you will be able to quickly determine the most suitable corrosion resistant material for your requirements. 240 pages. \$25

# HAIR PREPARATIONS 1969

by Alec Williams

Up-to-date technology of hair care products.

The rapidly rising demand for hair care preparations has resulted in a steady stream of new products for both the consumer and the professional beauty shop operator over the past few years. Active research and new product formulation have been carried out by a number of firms. This book discusses a significant portion of these later developments.

This book provides a detailed technological summary of recent developments based on 138 U.S. patents, since 1960, covering all aspects of hair preparations for the head, beard, eyelashes, and eyebrows.

Introduction, Dyeing, Bleaching, Waving, Setting, Shampoos-Rinses, Grooming-Tonics, Shaving Assistants, Other, Indexes. 208 pages, \$35



## FOOD GUIDE TO EUROPE 1969

This is a directory of the European Food Processing Industry that will help you in sales, market research, development, planning, acquisitions, and mergers. It is expected that the manufacture of processed foods in Europe will be a substantial growth industry.

Describes the 800 leading European food processing firms in 18 countries with this information (where available): Name and Address, Ownership, Plant Locations and Products, Local Subsidiaries and Affiliates, Foreign Subsidiaries and Affiliates, Principal Executives, Annual Sales, Number of Employees.

A valuable marketing guide. Only the 800 major food processing firms are included. This allows you to concentrate your efforts in the most profitable direction.

Will help you: Increase sales, Concentrate on the big buyers, Plan joint ventures, Make licensing arrangements, Know company officials, Prepare market research reports, Talk intelligently about the European food processing industry. 130 pages. \$20

## EUROPEAN FOOD PROCESSING INDUSTRY 1968

by S. A. Mann

This is a guide to the European Food Processing Industry that will help you in sales, market research, development, planning, acquisitions, and mergers. This is the first time that material of this nature has been published. There has been much information published on commodities, but very little on food processing, which is the subject of this report.

Western Europe is a long way behind the United States in the degree to which processed and convenience foods have been accepted. However, with the rapid increase in supermarkets and self-service stores, it is expected that the manufacture of processed and convenience foods will be a large growth industry in Europe.

For 18 countries, includes discussion, statistical material, and names and addresses of about 2,000 major food processors. 197 pages. \$35

## FREEZE DRYING OF FOODS AND BIOLOGICALS 1968

by Robert Noyes

*Food Processing Review No. 1*

The detailed, descriptive process information in this book is based on 105 U.S. patents in the freeze drying field—issued between January 1960 and May 1968. This book serves a double purpose in that it supplies detailed technical information, and can be used as a guide to the U.S. patent literature on freeze drying. By indicating only information that is significant, and eliminating much of the legal jargon in the patents.

Higher costs of freeze drying are due to high capital costs for equipment, as well as higher operating costs due to high energy consumption and more limited output. This book contains many cost-cutting ideas.

Since the products obtained by freeze drying are considerably superior in most cases than those produced by other processes, considerable research and development work has been carried out attempting to lower costs. This book will give you the latest developments and recent advances in freeze drying technology. Numerous illustrations. 313 pages. \$35.

## DEHYDRATION PROCESSES FOR CONVENIENCE FOODS 1969

by Robert Noyes

*Food Processing Review No. 2*

This is a large book in an 8 1/2 x 11 format. There are over 250,000 words describing 236 up-to-date dehydration processes for producing specific food products. This is the largest and most detailed body of information ever published on this subject.

The detailed, descriptive process information in this book is based on 236 U.S. patents in the food dehydration field—issued between January 1960 and May 1968. This book serves a double purpose in that it supplies detailed technical information, and can be used as a guide to the U.S. patent literature on dehydration of foods. By indicating only information that is significant, and eliminating much of the legal jargon in the patents; this book then becomes an advanced commercially oriented review of food dehydration processes.

Dry Milk Products, Cheese and Yogurt, Eggs, Fruit and Vegetable Juices, Fruits, Potatoes, Vegetables, Coffee, Tea, Miscellaneous. Many illustrations. 367 pages. \$35

## EDIBLE OILS AND FATS 1969

by Dr. N. E. Bednarczyk

*Food Processing Review No. 5*

This book describes in detail 225 recent process developments for producing edible oils and fats, based on the U.S. patent literature since 1960.

Shortenings; Fluid, Plastic, Miscellaneous: Margarine and Spreads; Margarine Oils, Highly Nutritional Oil Blends, Antispattering Agents, Fluid and Whipped Margarines, Flavor, Color, and Texture Modifications, Low Calorie Spreads; Salad Oils, Mayonnaise and Emulsified Dressings; Crystallization Inhibitors, Emulsified Dressings, Flavored Salad Oils, Low Calorie Dressings; Frying and Cooking Oils, Equipment, Breakdown Inhibitors, Antispattering Additives, Other Additives: Hard Butters; Preparation by Fractional Crystallization, Preparation by Ester Exchange, Miscellaneous: Oil Processing; Antioxidants and Stabilizers; Emulsifiers and Emulsions; Mixed Ester Emulsifiers, Dried Emulsion, Miscellaneous: Peanut Butter and Spreads; Chocolate Products; Indexes. Illustrations. 404 pages. \$35

## CONFECTIONARY PRODUCTS MANUFACTURING PROCESSES 1969

by Milton Gutterson

*Food Processing Review No. 6*

This book is of technological significance in that it details over 200 processes for producing confections, based on the U.S. patent literature since 1960.

Based solely on new technology, this book offers substantial manufacturing information relating to this field. The wide scope of detailed data can be seen by the chapter headings indicated below:

Candy  
Chocolate Products  
Whipped Products  
Icings  
Gels  
Coatings and Glazes  
Gums and Stabilizers  
Egg Products  
Marshmallows and Meringues  
Puddings  
Frozen Confections  
Chewing Gum  
Other Confections  
Indexes

Illustrations. 321 pages. \$35

## PROTEIN FOOD SUPPLEMENTS 1969

by Robert Noyes

*Food Processing Review No. 3*

The 126 Processes in this book are organized in 8 chapters by raw material source including the important newer processes for producing protein by fermentation of hydrocarbons. Another chapter on textured foods describes in detail a number of processes for producing these products that simulate meat. Indexes by company, inventors and patent number help in providing easily obtainable information.

This book is based upon the patent literature and serves a double purpose in that it supplies detailed technical information and can be used as a guide to the U.S. Patent literature on processes to obtain protein materials.

This book is an advanced commercially oriented technical review of processes to obtain protein materials. It includes the latest developments and recent advances in the field.

Contents: Hydrocarbon Fermentation, Fish-Based Protein, Soybeans, Cottonseed, Other Oilseeds and Legumes, Wheat and Gluten, Milk-Based Protein, Textured Foods, Miscellaneous, Indexes. Many illustrations. 412 pages. \$35

## SNACKS AND FRIED PRODUCTS 1969

by Dr. Alfred Lachmann

*Food Processing Review No. 4*

The sales of snack foods in the U.S. may reach the two billion dollar mark in 1969 according to the latest estimates. It is therefore not surprising that many companies are actively working on new snack foods or on improved processes. The patent literature on french fried potatoes, potato chips, corn chips and other crisps is continually growing and it is the purpose of this book to present this literature in easy readable form. In order to emphasize recent developments only those U.S. patents issued since 1960 are included.

French fried potatoes and their methods of production are described in the second chapter. The next chapter deals with potato chips, still the most popular product of the snack food industry. The U.S. market for potato chips is estimated to be approximately 600 million dollars in 1969. In Chapter Four the processes for corn chips are covered; in Chapter Five, apple crisps. Chapter Six describes processes for expanded chips and some specialty items; and the last chapter deals with batter mixes. Many illustrations. 181 pages. \$35

## VITAMIN B<sub>12</sub> MANUFACTURE 1969

by Robert Noyes

*Chemical Process Review No. 40*

Vitamin B<sub>12</sub> active substances are important therapeutic products for treatment of pernicious anemia. They have also been used for treatment of various other human ailments, and are also used as a veterinary growth factor. This book offers various methods of producing vitamin B<sub>12</sub> active substances.

Vitamin B<sub>12</sub> or cyanocobalamin is the most important of the vitamin B<sub>12</sub> active substances. An important analogue is hydroxocobalamin in which an OH replaces the CN group of the molecule.

The technical details outlined in this book are based on the U.S. patent literature. It is interesting to note that much of the basic work was done in the early 1950's. These patents are now just expiring, or will expire in the very near future.

Introduction, Streptomycin By-product, Chlorotetracycline By-product, Propionic Acid Bacteria to Produce Vitamin B<sub>12</sub>, Other Organisms to Produce Vitamin B<sub>12</sub>, Other Recovery and Purification Processes, From Sewage Sludge, Hydroxocobalamin, Other Active Materials and Compositions. 327 pages. \$35

## INORGANIC CHEMICAL AND METALLURGICAL PROCESS ENCYCLOPEDIA 1968

by Marshall Sittig

A single volume desk-top reference to inorganic chemical and metallurgical processes. Describes 882 processes—with 882 flow diagrams.

Today, there is more "glamour" in many inorganic chemicals and metals than there is in organic chemicals. For example, gallium arsenide, now important in the laser and semiconductor field was of no significance until only recently. The same can be said for many other inorganic and metallurgical chemicals that are now assuming importance due to rapidly changing developments in electronics, lasers, integrated circuits, cryogenics, superconductivity, materials development, nuclear energy, and the space program.

This book is organized in an unusual format. There is one inorganic chemical or metallurgical process on each page. At the top of the page an equipment drawing or flow diagram is shown, and underneath a description of the process is given. 883 pages—8 1/2" x 11"—hard cover. \$35

## AIR POLLUTION CONTROL PROCESSES AND EQUIPMENT 1968

by Marshall Sittig

*Chemical Process Review No. 24*

Air pollution is a technological problem, not only in the sense of research on pollutants, their nature, analysis, and elimination; but it is to a great extent application of known apparatus, processes, and concepts to environmental control.

Most complete descriptions of processes and equipment are available in the patent literature. These then, in most cases, cover processes and equipment which can be licensed or purchased today to combat today's and tomorrow's environmental control problems. Only through sound engineering principles in process design are we going to control air pollution at minimum cost. This book gives detailed process descriptions.

Contents: Sources of Air Pollution, Air Pollution Control Devices, Removing Specific Gases and Vapors from Air, Equipment for Removing Solids and Liquids from Air, Removing Specific Solids and Liquids from Air, Removing Automotive Exhaust Fumes from Air, Future Trends. 102 illustrations. 260 pages. \$35

## WATER POLLUTION CONTROL AND SOLID WASTES DISPOSAL 1969

by Marshall Sittig

*Chemical Process Review No. 32*

Describes specific water pollution processes, a substantial growth industry. Much of the technology necessary to effect water pollution control and solid wastes disposal has already been developed. In many cases these environmental problems could be resolved by intelligent process choice, and economic application of the proper control equipment.

This book in most cases covers processes and equipment which can be licensed or purchased today to combat today's and tomorrow's water pollution and solid waste disposal problems.

Introduction, The Water Problem, The Solid Waste Problem, Types of Water Contaminants, Removing Specific Inorganic Water Contaminants, Removing Specific Organic Water Contaminants, Removing Specific Solid Contaminants from Water, Handling Liquid and Solid Radioactive Wastes, Sewage Disposal, Solid Waste Disposal Processes, Future Trends. 78 illustrations. 244 pages. \$35



### NITRIC ACID TECHNOLOGY RECENT DEVELOPMENT 1969

by Dr. Robert Powell

*Chemical Process Review No. 30*

Introduction: Ammonia Oxidation Process, Wisconsin Thermal Process, Nitrogen Fixation by Shock Waves, Nitrogen Fixation in a Nuclear Reactor, Absorption of Nitrogen Oxides in Water to Form Nitric Acid Solution, Concentration of Dilute Nitric Acid Solutions, Direct Production of Concentrated Nitric Acid, Purification of Nitric Acid, Stabilizers for Nitric Acid. Numerous illustrations. 245 pages. \$35

### CITRIC ACID PRODUCTION PROCESSES 1969

by R. Noyes

*Chemical Processing Review No. 37*

Detailed descriptions of production processes for citric acid, based on the patent literature. Particular emphasis is placed on deep fermentation techniques. The use of citric acid as an acidulant in the food industry will increase, even though other acids are making inroads in citric acid consumption. It is expected that the use of citric acid in non-food industries will increase in the future. 157 pages. \$24

### FERTILIZER DEVELOPMENTS AND TRENDS 1968

by A. V. Slack

This book analyzes the opportunities for improving fertilizer technology based on current world-wide trends in research and development.

A basic assumption of the book is that the reader is familiar with the fertilizer field, and chemistry of fertilizers and current manufacturing methods are discussed only to the extent necessary for adequately relating new departures to standard practice. Moreover, older developments have been included only when necessary for an adequate description of the newer ones—the major emphasis is in departures that have been introduced in the past five or six years, and new developments that will assume major importance in the future.

Contents: Research and Development, Ammonia, Ammonium Nitrate, Urea, Ammonium Sulfate, Slow Release Nitrogen, Other Nitrogen Fertilizers, Phosphoric Acid, Ammonium Phosphate, Nitric Phosphates, Superphosphate, The mal and Miscellaneous Phosphate Processes, Potassium Fertilizers, Fluid Fertilizers, Bulk Blending, Minor Nutrients. 98 illustrations, 406 pages. \$35

### NEW FERTILIZER MATERIALS 1968

*Centre International  
des Engrais Chimique (C. I. E. C.)*

This book is invaluable to fertilizer and chemical manufacturers who are looking to produce or market potentially useful new fertilizer materials. The agronomist and other specialists in this field will find the book to be extremely useful. The book contains extensive technical, economic, and agronomic information.

Ureaform, Crotonylidenediurea (CDU), Isobutylidene Diurea, Superphosphoric Acid, Triple Superphosphate of 55%  $P_2O_5$  Content, Ammonium Phosphates, Ammonium Polyphosphate, Nitrophosphates, Nitrate of Potash, Potassium Metaphosphate (Potassium Polyphosphate), Monopotassium Phosphate and Monoammonium Phosphate, Magnesium as a Plant Nutrient, Magnesium Phosphates, Magnesium Ammonium Phosphate and Related Compounds, Sulfur as a Fertilizer, Oxamide, Urea Nitrate, Urea Phosphate, The Hydrides of Phosphorus, Red Phosphorus, Fertilizer Application Equipment, Modern Use of Fertilizer. Numerous illustrations. 430 pages. \$35

### PESTICIDE PRODUCTION PROCESSES 1967

by Marshall Sittig

*Chemical Process Review No. 5*

The pesticide industry in the United States is expected to grow to a one billion dollar industry by 1975, and a two billion dollar industry by 1985 (at manufacturers' price levels). Worldwide growth of pesticides will be even faster, due primarily to rapidly increasing local food production in the developing countries.

This book details specific manufacturing processes for the major commercial pesticides (insecticides, fungicides, and herbicides). Included is such information as: Feed Materials; Temperature; Pressure; Reaction Time; Reaction Medium; Catalysts; Reactor Design; Product Recovery.

Production processes for these pesticides are described: Olefin-based Insecticides; Olefin-based Fungicides; Olefin-based Herbicides; Diolefin-based Insecticides; Diolefin-based Fungicides; Diolefin-based Herbicides; Aromatic-based Insecticides; Aromatic-based Fungicides; Aromatic-based Herbicides. 48 flow diagrams. 200 pages. \$35.

### CONTROLLED RELEASE FERTILIZERS 1968

by Dr. Robert Powell

*Chemical Process Review No. 15*

This book offers you complete technical data on numerous processes and products in this field. The two major approaches are (a) compounds of low solubility, and (b) coated granules.

Introduction; Compounds of Low Solubility, Coated Granules, Prevention of Nitrogen Losses, Rapid-Release Fertilizer. 279 pages. \$35

### AMMONIUM PHOSPHATES 1969

by Dr. M. W. Ranney

*Chemical Processing Review No. 35*

This book describes recent processes for production of ammonium phosphates. The 63 processes are based on U.S. patents issued since 1960 and offer a comprehensive treatment of up-to-date technical information.

Introduction; Ammonium Orthophosphates, Diammonium Orthophosphates, Ammonium Polyphosphates, Metal Ammonium Phosphates, Ammonium Phosphate—Ammonium Nitrate Mixtures.

Many illustrations. 278 pages. \$35

### FERTILIZER INDUSTRIES OF EUROPE 1968

by the British Sulphur Corporation

Contains this information for each Country: General statistics, Introduction, Fertilizer Production, Fertilizer consumption, Fertilizer Exports/Imports, Producers (Names and addresses of the major producers with a discussion of their activities), Graph showing fertilizer consumption and agricultural production from 1962 through 1967.

A pertinent summary of the European fertilizer industry. 124 pages. \$35

### UREA PROCESS TECHNOLOGY 1968

by Dr. Robert Powell

*Chemical Process Review No. 13*

The information in this book is up-to-date, and is based on information made available since 1960. The information in this volume includes a huge variety of numerical data. Also included are 67 flow diagrams or equipment illustrations. The bibliography contains 142 references.

General Considerations, Processes, Low Biuret Processes, Product Finishing and Controlled Release Fertilizer. 324 pages. \$35

### PHOSPHORIC ACID BY THE WET PROCESS 1967

by Robert Noyes

*Chemical Process Review No. 9*

This book is a practical publication devoted to production of phosphoric acid by the wet process. Production technology is changing rapidly, with new single tank reactors, the advent of superphosphoric acid, new commercial processes for acidulation with hydrochloric and nitric acids, new semihydrate and anhydrite processes, etc. This book describes these as well as conventional processes, in detail. 282 pages. \$35

### AMMONIA AND SYNTHESIS GAS 1967

by Robert Noyes

*Chemical Process Monograph No. 26*

Outlines technology and economics pertaining to ammonia production, with many flow diagrams and tables. The discussion is organized in reference form, beginning with the various methods of hydrogen and synthesis gas preparation, shift conversion techniques, various carbon dioxide removal systems, final purification, compression, and ammonia synthesis. Bibliography. Many illustrations. 175 pages. \$20

### POTASH AND POTASSIUM FERTILIZERS 1966

by Robert Noyes

*Chemical Process Monograph No. 15*

1. Introduction, Markets, Future Projections, 2. Geology and Geography, 3. Underground Deposits of Soluble Minerals, 4. Brines, 5. Other Sources of Potash, 6. Flotation, 7. Crystallization, 8. Other Refining Processes, 9. Potassium Sulphate, 10. Potassium Nitrate, 11. Potassium Phosphates, 12. Final Product. 210 pages. \$15

### EUROPEAN PHARMACEUTICAL MARKET REPORT 1967

by S. A. Mann

This book has been prepared to help you obtain a "birds-eye-view" of the European pharmaceutical industry. Gives a variety of market research data.

General and Historical Background, Foreign Trade, Distribution, Market Structure, Structure of the Industry, Names and Addresses of Major Firms, Growth of the Industry, Prices, Research and Development, Regulations and Development, Health Services. 111 pages. \$35



10 270

To be issued from 26th Nov., 1970

CHECKED  
2008

VERIFIED  
2013

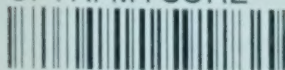
|   |         |
|---|---------|
| V.M. Mandotkrot<br>P.T.                               |         |
| <del>Dr. (Mrs) Usha V. Mandotkrot</del><br>(M)        |         |
| <del>P. 30/11</del><br><del>N. Subramanian (PT)</del> |         |
| <del>H. K. Uts (PT)</del>                             |         |
| Ref: 196  |         |
| 29.5.90   | 15.6.90 |
| 30.6.90   | 10/8    |

as expended to obtain each drug. There is no information is listed. Most of obtained from numerous sources, the literature contains due to typographical was required to obtain these drugs where patent from the literature, the directly from the producer or thousands of dollars spent.

tents directly from the  
st you \$170.50 for the  
required to place the  
e \$95.50 by purchasing

**1705 pages—3 bindings—\$75**

CFTRI-MYSORE



9588  
Animal feeds 197.



## FOOD PROCESSING REVIEWS

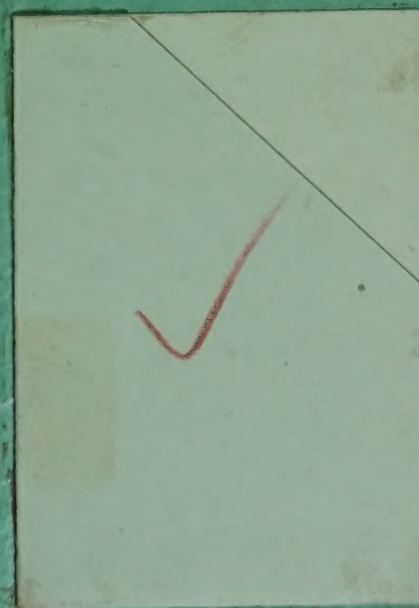
Food Processing Reviews are a series of books outlining up-to-date food manufacturing technology. They relate to plant production processes for producing food and beverage products.

|  |      |
|--|------|
| Freeze Drying of Foods and Biologicals; Noyes - \$35               | 1968 |
| Dehydration Processes for Convenience Foods; Noyes - \$35          | 1969 |
| Protein Food Supplements; Noyes - \$35                             | 1969 |
| Snacks and Fried Products; Lachmann - \$35                         | 1969 |
| Edible Oils and Fats; Bednarczyk - \$35                            | 1969 |
| Confectionary Products - Manufacturing Processes; Gutterson - \$35 | 1969 |
| Alcoholic Malt Beverages; Gutcho - \$35                            | 1969 |
| Baked Goods; Gutterson - \$35                                      | 1969 |

## ELECTRONICS MATERIALS REVIEWS

Electronics Materials Reviews are a series of comprehensive up-to-date studies of the technology of production and utilization of the materials used in electronic devices. They are written from the practical processing point of view giving specific production technology, materials characteristics, construction of equipment, and operating conditions.

|  |      |
|--|------|
| Pure Chemical Elements for Semiconductors; Sittig - \$35   | 1969 |
| Manufacture of Semiconductor Compounds; Sittig - \$35      | 1969 |
| Semiconductor Crystal Manufacture; Sittig - \$35           | 1969 |
| Doping and Semiconductor Junction Formation; Sittig - \$35 | 1970 |
| Producing Films of Electronic Materials; Sittig - \$35     | 1970 |





ndc